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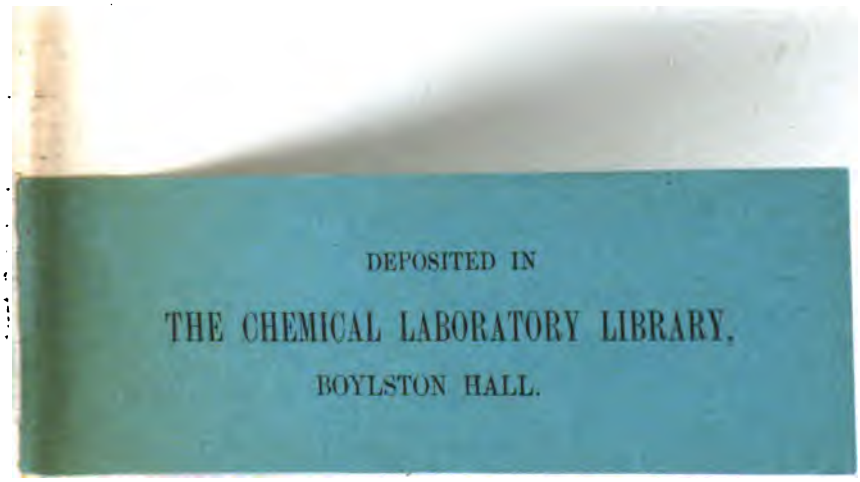

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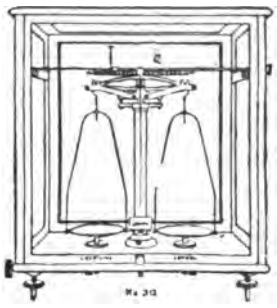

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NOTE ON THE QUANTITATIVE ESTIMATION OF LEAD IN ALLOYS.

By W. ELBORNE, M.A., and C. M. WARREN.

THE methods described in modern text-books on quantitative analysis for the estimation of lead in alloys, such as stereo and type metals, are exceedingly tedious and cumbersome; for instance, one of the methods advocated, operating upon 1 grm. of type metal, involves the use of aqua regia, 10 grms. of tartaric acid, 35 grms. of caustic soda dissolved in 500 cc. of water, boiling, saturating with sulphuretted hydrogen, filtering, boiling the precipitated sulphides in nitric acid till syrupy, diluting with water again, and filtering off traces of tin, evaporating with sulphuric acid, washing with alcohol, filtering, drying, igniting, and weighing as lead sulphate.

The process which we now introduce will be found to be very simple and to give quite as accurate results, and is applicable to all alloys of lead which are soluble in the solvent used. The principle of the process is based on the insolubility of lead chloride in absolute alcohol, whereas the chlorides of antimony, tin, arsenic, bismuth, iron, nickel, cobalt, manganese, copper, zinc, cadmium, aluminium, and magnesium are all soluble.

The process consists in taking 1 grm. of the alloy in fine shavings, placing it in a flask, and adding about 50 cc. of concentrated hydrochloric acid, then passing a slow current of chlorine gas through the liquid, in the cold, for some twenty-four hours; this is readily effected by adding some peroxide of manganese to concentrated hydrochloric acid in the cold in another flask, when, without application of heat, sufficient chlorine will be found to be continuously evolved, which is allowed to bubble through the liquid; after three or four agitations of the flask during the twenty-four hours, the whole of the alloy (stereo metal) will have passed into solution; in other more refractory alloys after saturation with chlorine the solution may be boiled, and the operation repeated until all is dissolved. When solution of the alloy is complete, transfer the liquid in small quantities at a time to a small weighed porcelain dish, and evaporate to dryness on a water-bath; allow to cool in a desiccator, then flood the dry residue with absolute alcohol, quickly stir with a glass rod, and immediately pour off the supernatant liquid through a dry weighed filter-paper; repeat tnis operation of washing with alcohol and filtration three or four times, allowing the last lot of alcohol to remain on the residue in the dish for five minutes before pouring off. Finally, transfer the dish with its contents, also the filter-paper, to a water-oven, and dry at 100° C. for three hours. Weigh as lead chloride, $PbCl_2$. The following is a specimen of the results obtained by the above process by merely pouring off the alcohol without any filtration, the slight deficiency in the amount of lead found being due to a small quantity of lead chloride poured off with the alcohol.

Known composition of alloy (grm.)	Lead	0·810	1·000
	Antimony .	0·150	
	Tin	0·040	
Lead chloride (grm.)			1·085
Equivalent to lead (grm.)			0·808

STUDIES OF THE PROCESSES OPERATIVE IN SOLUTIONS.*

By H. E. ARMSTRONG, F.R.S.

PART VI.—HYDROLYSIS, HYDROLATION AND HYDRONATION AS DETERMINANTS OF THE PROPERTIES OF AQUEOUS SOLUTIONS.

THE paper contains an account of a theory in explanation of the properties of solutions. Water is regarded as a complex mixture containing molecules of a variety of types; it is proposed to confine the term water to the mixture. It is argued that the active molecules in water are monad *hydrone* (H_2O) molecules and *hydrol-hydrone* (briefly *hydrol*) molecules, $H_2O{<}^{H}_{OH}$; the inactive, a series of polyhydrones of the form—

$$H_2O{:}OH_2, \qquad \begin{matrix} H_2O{-\!-}OH_2 \\ \diagdown\ \diagup \\ OH_2 \end{matrix}, \text{ \&c.}$$

When a substance RX is dissolved, if a non-electrolyte, in part it undergoes conversion into a hydrol $RX{<}^{H}_{OH}$; if capable of forming a conducting solution, it is not only hydrolated but itself in part *distributed* and converted into a hydrone of the type $H_2O{<}^{R}_{X}$: the mixture of these two isodynamic forms in solution constitutes a composite electrolyte, and electrolysis is the outcome of their reciprocal interaction under the influence of the current. The changes which take place in aqueous solution involve the interaction of such composite molecules—for example, when an acid is neutralised by an alkali—$HCl{<}^{H}_{OH} + NaHO{<}^{OH}_{H} = NaCl + 3H_2O$; the hydrol thus displaced forms water of the composition appropriate to the conditions prevailing in the solution. In this manner

* Abstract of a Paper read before the Royal Society, June 18th, 1908.

the large expansion is accounted for which attends the neutralisation of an acid by alkali. It is shown that the process is generally applicable.

Substances may also exist in solution in the form of *hydrones*, *e.g.*, $NaCl : OH_2$—in such compounds the water probably has greater optical effect than ordinary water: hence the increase of refractive power which in many cases attends dissolution in water.

THE REDUCTION OF VANADIC ACID BY ZINC AND MAGNESIUM.

By F. A. GOOCH and GRAHAM EDGAR.

A METHOD recently proposed by B. Glasmann (*Ber.*, xxxviii., 600) for the estimation of vanadic acid and molybdic acid in association with one another, depends upon the dissimilarity in the action of zinc and magnesium upon the former and the similarity in the action of these metals upon the latter in the presence of hydrochloric acid. The method consists essentially in treating for an hour to an hour and a-half, in a flask closed with a Bunsen valve, each of two aliquot portions of the solution containing a vanadate and a molybdate, the one with zinc and hydrochloric acid under gentle heating and the other with magnesium and hydrochloric acid, opening the flasks to the air, pouring the contents of each flask into a 500 cc. porcelain dish containing a solution of 10 grms. of manganese sulphate in 300 cc. of air-free boiling-hot water, and titrating with potassium permanganate while stirring actively. The amount of permanganate taken in titrating the contents of the first flask is used presumably in oxidising the molybdenum from the condition of Mo_2O_3 to that of MoO_3 and the vanadium from the condition of V_2O_2 to that of V_2O_5: the amount of permanganate used in titrating the contents of the second flask shows presumably oxidation corresponding to a change of Mo_2O_3 to MoO_3 and to a change of V_2O_2 to V_2O_5. The difference between the amounts of permanganate used in the two titrations should presumably indicate the oxidation of V_2O_2 to V_2O_3 and so by calculation give the amount of V_2O_5 originally present in the solution. The amount of V_2O_5 having been determined, the amount of MoO_3 originally present is easily calculated from the amount of permanganate used in either titration.

It is plain that the value of the method depends largely upon the definiteness with which vanadic acid may be reduced to the condition of V_2O_3 by magnesium and left in the condition of V_2O_2 when the solution is treated with zinc and subsequently exposed to the air.

Concerning the reduction of vanadic acid by magnesium in the presence of hydrochloric acid, Glasmann gives the data and results of three experiments (Titrations Tabelle II., *loc. cit.*). Of these two are in fair agreement with the hypothesis; but, unfortunately, the record of the third experiment is affected by an error of about 10 per cent, either typographical or of calculation. It is likewise interesting to note, though the conditions are different, that in Roscoe's study of the behaviour of vanadic acid in the presence of magnesium and sulphuric acid (*Ann. Chem. Suppl.*, 1868, vi., 77), to which Glasmann refers, the variation in the degree of reduction is likewise considerable, amounting to more than 2 per cent.

As to the reduction of vanadic acid by zinc, Roscoe's results show a degree of reduction approaching that of V_2O_2, but with variations of as much as 3 per cent; and it is well known that the oxidation of solutions thus reduced proceeds rapidly and ultimately results in the condition represented by V_2O_4 (Roscoe, *loc. cit.*; Gooch and Gilbert, *Am. Journ. Sci.*, xv., 390). Whether reductions may be kept regular and the action of air made so small as not to vitiate the analytical results of Glasmann's method during the short exposure, are matters for experimental determination. According to Glasmann's results in these experiments, the interference would seem to be inappreciable, but here again the calculated results are not in complete accord with the printed data (Titrations Tabelle I., *loc. cit.*).

It has seemed desirable, therefore, to consider further the accuracy which may be expected under the conditions of analysis in the reduction of vanadic acid by magnesium and by zinc.

Reduction by Magnesium.

Portions of a solution of sodium vanadate, standardised by the very accurate method of Holverscheit, were reduced by magnesium in the presence of hydrochloric acid or sulphuric acid (Inaugural Dissertation, Berlin, 1890).

In each of the experiments of Table I., A, the reduction was made, in a funnel-trapped flask, by magnesium in the presence of hydrochloric acid added gradually to properly moderate the action. After the magnesium had been used up, the contents of the flask were added to 800 cc. of boiled water containing 1 grm. of manganese sulphate and 2 cc. of sulphuric acid, and titration was made with nearly N/10 potassium permanganate. The conditions of the experiment were essentially those of Glasmann's method in respect to reagents and time of action.

In the experiments of Table I., B, the treatment was modified so that the action might be slower and long continued, with a view to bringing about better contact of the vanadium solution with the magnesium cushioned by evolved hydrogen. The reduction was made in a stoppered flask connected with a hydrogen generator, arranged for the maintenance of an atmosphere of hydrogen during the period of reduction, and fitted with a separatory funnel through which dilute sulphuric acid was added from time to time in small portions as required during the prolonged action, never evolving hydrogen violently enough to float the magnesium.

The experiments of Table I., C, were made to test the effect of using magnesium in the form of a heavy amalgam. About 2 grms. of magnesium were dissolved, with heating, in an excess of mercury, and the resulting liquid amalgam was used as the reducing agent, in the flask containing vanadic acid and sulphuric acid. At the end of the action, which took place without the application of heat and was over in about ten minutes, the distinctly violet solution was decanted, filtered from the mercury with careful washing, and titrated with potassium permanganate. Special tests in blank having shown that, for the amount of amalgam used, mercury goes into solution and acts upon the permanganate sufficiently to use up about 0·7 cc. of that reagent, a deduction of that amount from the total amount used was made for such action in each experiment, before calculating the degree to which the vanadic acid had been reduced.

From these results it is plain that the degree in which vanadic acid may be reduced by magnesium in the presence of hydrochloric acid or sulphuric acid is irregular and dependent upon conditions not easily controlled. With magnesium amalgam the reduction proceeds most readily and approximates more or less to the condition of V_2O_2.

It is obvious that under none of the conditions which we have tried is the reduction by magnesium sufficiently definite to be applied in a good analytical process.

In Table II. are given the results of the reduction of vanadic acid by zinc. The details of experiments made in the reduction flask with zinc and hydrochloric acid are given in A; the record of experiments also made in the reduction flask with zinc and sulphuric acid is given in B; and C contains the data of experiments made with the Jones reductor, in which a column of amalgamated zinc 40 cm. long and 2 cm. in diameter was used (Blair, "The Chemical Analysis of Iron," 6th Ed., 225). In all these experiments the titration was effected directly in air by nearly N/10 potassium permanganate. The V_2O_5 is calculated upon the hypothesis that the reduction goes to the condition of V_2O_2.

From the results of Table II. it is evident that in no case does the condition of oxidation of the product of the

Table I.

Time.	Magnesium met. Grms.	$KMnO_4$ N/10 × 1·052. Cc.	V_2O_5 taken as $NaVO_3$. Grm.	V_2O_5 calculated from oxidation of V_2O_3. Grm.	Error in terms of V_2O_5. Grm.	V_2O_3 reduced to— V_2O_3. Grm.	V_2O_2. Grm.
			A.—*Reduced by Magnesium and Hydrochloric Acid.*				
1 hour	4·0	26·05	0·1144	0·1249	+0·0105	0·0934	0·0210
1 „	4·0	26·2	0·1144	0·1257	+0·0113	0·0918	0·0226
1½ „	4·0	27·1	0·1144	0·1300	+0·0156	0·0834	0·0312
1¾ „	4·0	28·3	0·1144	0·1357	+0·0213	0·0718	0·0426
			B.—*Reduced by Magnesium and Sulphuric Acid.*				
8 hours	4·0	24·1	0·1144	0·1156	+0·0012	0·1120	0·0024
8 „	4·0	23·8	0·1144	0·1142	−0·0002	—	—
8 „	4·0	25·2	0·1144	0·1208	+0·0064	0·1015	0·0129
8 „	4·0	25·4	0·1144	0·1218	+0·0074	0·0996	0·0148
16 „	8·0	26·5	0·1144	0·1271	+0·0127	0·0890	0·0254
16 „	8·0	26·1	0·1144	0·1252	+0·0108	0·0928	0·0216
20 „	12·0	26·8	0·1144	0·1285	+0·0141	0·0861	0·0283
			C.—*Reduced by Magnesium Amalgam and Sulphuric Acid.*				
				Calculated from V_2O_3.			
10 mins.	2·0	32·1	0·1144	0·1027	−0·0117	0·0353	0·0791
10 „	2·0	33·4	0·1144	0·1068	−0·0076	0·0228	0·0916
10 „	2·0	34·1	0·1144	0·1090	−0·0054	0·0161	0·0983
10 „	2·0	32·4	0·1144	0·1034	−0·0110	0·0329	0·0815
10 „	2·0	33·1	0·1144	0·1054	−0·0090	0·0269	0·0875
10 „	2·0	33·6	0·1144	0·1075	−0·0069	0·0208	0·0936

Table II.

Experiment.	$KMnO_4$. N/10 × 1·095. Cc.	V_2O_5 taken as $NaVO_3$. Grm.	V_2O_5 calculated from oxidation of V_2O_3. Grm.	Error in terms of V_2O_5. Grm.	Remarks.
		A.—*Reduced in Flask with Zinc and Hydrochloric Acid.*			
1.	35·0	0·1235	0·1166	−0·0069	Poured out into dish; titrated in presence of $MnSO_4$.
2.	35·85	0·1235	0·1194	−0·0041	
3.	34·9	0·1235	0·1163	−0·0072	
		B.—*Reduced in Flask with Zinc and Sulphuric Acid.*			
	N/10 × 1·0956.				
4.	36·3	0·1235	0·1203	−0·0032	Titrated in the reduction flask.
5.	36·4	0·1235	0·1209	−0·0026	
6.	36·2	0·1235	0·1213	−0·0022	
7.	36·05	0·1235	0·1206	−0·0029	
8.	36·1	0·1235	0·1201	−0·0034	
9.	35·68	0·1235	0·1188	−0·0047	Poured into dish for titration.
10.	34·8	0·1235	0·1159	−0·0076	Exposed in dish 20 minutes.
		C.—*Reduced by column of Amalgamated Zinc.*			
	N/10 × 1·052.				
1.	42·4	0·1381	0·1356	−0·0025	Titrated in the receiver exposed to the air.
2.	42·7	0·1381	0·1366	−0·0015	
3.	42·2	0·1381	0·1350	−0·0031	
4.	42·5	0·1381	0·1359	−0·0022	

Table III.

Ferric alum, 10 per cent sol. Cc.	Phosphoric acid, syrup. Cc.	$KMnO_4$. N/10 × 1·052. Cc.	V_2O_5 taken as $NaVO_3$. Grm.	V_2O_5 calculated from oxidation from V_2O_3. Grm.	Error in terms of V_2O_5 Grm.
25	5	43·10	0·1381	0·1378	−0·0003
25	5	43·20	0·1381	0·1381	—
25	5	43·30	0·1381	0·1384	+0·0003
25	5	43·28	0·1381	0·1384	+0·0003
25	5	43·32	0·1381	0·1385	+0·0004
15	3	21·60	0·0691	0·0690	−0·0001
15	3	21·62	0·0691	0·0691	—
15	3	21·80	0·0691	0·0696	+0·0005
40	8	64·90	0·2072	0·2075	+0·0003

reduction of vanadic acid by zinc and hydrochloric acid in the flask, by zinc and sulphuric acid in the flask, or by amalgamated zinc in the reductor, correspond exactly to V_2O_2 when titration is made in air. A series of experiments was made, therefore, in which care was taken to protect the reduced solution from the action of air until the highest degree of oxidation is past and registered, by an adaptation of a method used by Randall (*Am. Journ. Sci.*, xxiv., 313) for the accurate titration of molybdic acid similarly reduced by the column of amalgamated zinc. (We are indebted to Professor Henry Fay for the information that this mode of treating reduced molybdic oxide was first worked out many years ago by Dr. C. B. Dudley, though never published). According to this procedure, the receiver attached to the zinc column was charged with a solution of ferric alum, gentle suction was applied to the receiver, and in succession were passed through the column of amalgamated zinc, hot water (100 cc.), 2·5 per cent sulphuric acid (100 cc.), the solution of vanadic acid in 2·5 per cent sulphuric acid (125 cc.), and finally hot water (200 cc.). To the receiver was added syrupy phosphoric acid (4 cc.), to decolorise the solution, and the titration was made in the hot solution in the usual manner with potassium permanganate. Table III. contains the results of these experiments, corrected for the action of the zinc column upon the reagents without the vanadic acid.

The results of Table III. show plainly that the action of the column of amalgamated zinc carries the reduction easily and rapidly to the condition of V_2O_2, and that by anticipating the oxidising action of the air by means of a ferric salt in the receiver the solution is made less sensitive to the action of the air, while the highest degree of reduction is registered by the ferrous salt formed. A comparison of these results with those of Table II. shows unmistakably that a solution containing vanadium in the condition of V_2O_2 cannot be exposed to air, even momentarily, without undergoing oxidation.

Finally, it is evident that the assumptions upon which Glasmann's method for the determination of vanadic and molybdic acids is based—viz., that the condition of vanadium in a solution reduced by magnesium is definitely that of V_2O_3 and that the condition of vanadium in a solution reduced by zinc and exposed subsequently to the air is definitely that of V_2O_2—are unwarranted. — *American Journal of Science*, [4], xxv., p. 233.

THE ACTION OF VARIOUS ANHYDROUS CHLORIDES ON TELLURIUM AND ON TELLURIUM DIOXIDE.

By VICTOR LENHER.

In an earlier paper (*Journ. Am. Chem. Soc.*, xxiv., 188) the action of sulphur monochloride on elementary tellurium has been shown to result in the production of tellurium tetrachloride and sulphur, when the sulphur monochloride is in excess, while Krafft and Steiner (*Ber.*, xxxiv., 560), in studying this reaction, observed that when an excess of tellurium is heated with sulphur monochloride, tellurium dichloride results.

Further study on the action of tellurium and the dioxide with active reagents has shown that with many of the anhydrous chlorides, especially with those which are liquid at the ordinary temperature, tellurium tetrachloride is produced. In certain cases the tetrachloride immediately separates from the solution in pure form, while with a number of reagents of this character actual union takes place, and a crystalline condensation product separates.

Tellurium Dioxide and Sulphur Monochloride.—Tellurium dioxide, when treated with an excess of sulphur monochloride, is transformed into the tetrachloride, sulphur dioxide being formed at the same time, according to the equation $TeO_2+2S_2Cl_2=TeCl_4+SO_2+3S$.

Analysis, $TeCl_4$.—Calculated—Cl, 52·79; Te, 47·21. Found—Cl, 52·07; Te, 46·65.

When, on the other hand, an excess of tellurium dioxide is heated with sulphur monochloride, the reducing action of this reagent steps in, and the result is that the dichloride of tellurium is formed, thus:— $TeO_2+S_2Cl_2=TeCl_2+S+SO_2$.

The formation of tellurium tetrachloride by the action of excess of sulphur monochloride on tellurium dioxide takes place readily; the reaction can be materially hastened by warming, and under these conditions, preparation of a large amount of the tetrachloride can be accomplished in a very short time. Extraction of the salt with carbon bisulphide is advisable in order to remove an excess of sulphur.

Behaviour of the Oxychloride of Sulphur toward Tellurium and Tellurium Dioxide.—With thionyl chloride, either the oxide of tellurium or the metal yields tetrachloride when the anhydrous chloride is in excess, and dichloride when the element or oxide is in excess. These reactions are quite similar to the action of sulphur monochloride on the metal or oxide.

Sulphuryl chloride reacts with the element forming tellurium tetrachloride, but when the metal is in excess the dichloride is the resulting product. Tellurium dioxide in contact with sulphuryl chloride acts only very slowly, if at all, in the cold, but when the two are heated together in a sealed tube, crystalline products are formed which, by analysis, appear to be due to the simple solution of the dioxide in the sulphuryl chloride and subsequent recrystallisation of condensation products. The end products of the reaction are dependent on the reacting masses, and on the pressure at which the reaction is carried out.

Tellurium and Thionyl Chloride (with C. W. Hill).—Tellurium and thionyl chloride, when heated together in a sealed tube for twenty-four hours, yield crystals of the tetrachloride. The supernatant liquid was dark coloured, and was found to contain tellurium.

Analysis, $TeCl_4$.—Calculated—Cl, 52·79; Te, 47·21. Found—Cl, 52·00; Te, 47·47, 46·60.

In studying the action of thionyl chloride on tellurium it was found most satisfactory to heat the metal in a porcelain boat in a current of the vapour of thionyl chloride, which was produced by boiling thionyl chloride in a distilling bulb. The boat in which the tellurium was contained was placed in a combustion tube connected directly with the distillation bulb, and the combustion tube was drawn out into a series of bulbs. This apparatus was chosen inasmuch as the arrangement allowed the products of the reaction, by carefully regulating the temperature, to be carried from bulb to bulb, and in the first bulbs practically all of the sulphur formed in the reaction was retained, allowing the tellurium tetrachloride to be collected in the bulbs farthest from the boat.

Analysis, $TeCl_4$.—Calculated—Cl, 52·79; Te, 47·21. Found—Cl, 52·80, 52·20; Te, 47·01, 46·95.

The reaction of thionyl chloride on tellurium may be expressed, $2SOCl_2+Te=TeCl_4+SO_2+S$.

Thionyl Chloride and Tellurium Dioxide (with C. W. Hill).—By heating thionyl chloride with tellurium dioxide for twenty-four hours in a sealed tube, long slender crystals of tellurium tetrachloride were obtained. When the tubes in which the reaction was carried out were opened, sulphur dioxide was liberated.

Analysis, $TeCl_4$.—Calculated—Cl, 52·79; Te, 47·21. Found (I.)—Cl, 52·31, 52·71; Te, 47·11, 47·64. (II.)—Cl, 52·55, 52·13; Te, 47·55, 47·20.

On heating the dioxide in the vapour of thionyl chloride, tellurium tetrachloride is also readily obtained. Analysis II., above.

Sulphuryl Chloride and Tellurium (with C. W. Hill).—Tellurium and sulphuryl chloride, when heated together in a sealed tube, yield tellurium tetrachloride. On heating

tellurium in the vapour of sulphuryl chloride, the metal is first transformed into black dichloride, after which it is further chlorinated to the tetrachloride by the action of excess of the sulphuryl chloride.

Analysis, $TeCl_4$.—Calculated—Cl, 52·79; Te, 47·21. Found—Cl, 52·51, 52·36; Te, 47·14, 47·30.

Sulphuryl Chloride and Tellurium Dioxide (with C. W. Hill).—Tellurium dioxide acts quite differently toward sulphuryl chloride than it does with thionyl chloride or sulphur monochloride. When heated in the vapour of sulphuryl chloride to as high a temperature as it is possible to conduct an experiment in hard glass combustion tubing, there is apparently no action. No volatilisation of the tetrachloride occurs, and in our experiments, when a weighed amount of tellurium dioxide was heated in the vapour of sulphuryl chloride to as high a temperature as the glass would permit, the boat and contents weighed precisely the same after this treatment as before. This would indicate that under these conditions the vapour of sulphuryl chloride is without any appreciable action on tellurium dioxide.

When, however, tellurium dioxide and sulphuryl chloride are brought together in a sealed tube and heated, the system undergoes complex changes. When the dioxide was heated in a sealed tube with sulphuryl chloride for twenty-four hours at 165°, a small amount of a crystalline substance was formed, but the larger part of the dioxide remained unchanged. The heating was continued for seventy-two hours, when the dioxide completely disappeared, the liquid having assumed a dark yellow colour. On allowing the tube to cool, crystals deposited which were entirely unlike tellurium tetrachloride. When the tube was opened so much sulphur dioxide had been formed that the major part of the contents were lost when the pressure was released on opening the tube. The portion saved showed a content of 40·38 per cent of chlorine.

The experiment was repeated a number of times, the tubes being heated for forty-eight hours to 175°. In each case the tube was strongly cooled before opening, and in each tube a crystalline product was obtained.

Analysis showed the crystalline substances formed to vary in composition. Their composition by the analytical data obtained may be indicated as follows:—$3TeO_2.4SO_2Cl_2$; $5TeO_2,9SO_2Cl_2$; $TeO_2,2SO_2Cl_2$; $2TeO_2.5SO_2Cl_2$.

Sulphuryl chloride appears to have no action on tellurium dioxide at the ordinary temperatures, or when the dioxide is heated in the vapour of sulphuryl chloride, but when the two are heated together to high temperatures in a sealed tube, condensation products of varying composition are obtained.

In each experiment it was found that the excess of sulphuryl chloride contained large quantities of tellurium.

General Remarks.

In studying the action of the chloride of sulphur or the oxychlorides of sulphur it is observed that not infrequently one of the products of the reaction along with tellurium chloride is sulphur dioxide. Frequently in our experiments, in sealed tubes, large volumes of sulphur dioxide escaped on opening the tubes, while in the tube itself pure white tellurium chloride remain. This is an interesting illustration of mass action, as well as of conditions in the system, since sulphur dioxide readily precipitates tellurium from an aqueous or acid solution of the chloride, and the dioxide when heated in a current of sulphur dioxide gas is readily reduced to metal.

While the action of the various chlorides of sulphur on tellurium or on tellurium dioxide goes on readily, a number of other anhydrous chlorides act similarly, but not infrequently by-products are formed which contaminate the chloride of tellurium which is produced. For example, *arsenic trichloride* or *antimony trichloride* react with tellurium dioxide, yielding tellurium tetrachloride and arsenic trioxide, or antimony trioxide. In these cases it is very difficult to obtain the tellurium chloride in a high degree of purity. *Lead tetrachloride* will convert either the element or the oxide to tellurium tetrachloride. Here, again, an impure salt is formed, and a mixture is obtained from which it is difficult to obtain pure tellurium salt.

Phosphorus trichloride, in contact with the dioxide of tellurium, quickly reduces it to the elementary condition, and when brought in contact with the element in pure condition is without action.

Carbon tetrachloride is without action on tellurium or on tellurium dioxide even when allowed to be in contact with either of them for a great length of time.

Tellurium Dioxide and Antimony Pentachloride.—Antimony pentachloride reacts with tellurium dioxide in the cold, the action of heat materially hastening the reaction, with the formation of the compound $TeCl_4SbCl_5$.

Analysis, $TeCl_4.SbCl_5$.—Calculated—Te, 22·49; Sb, 21·16; Cl, 56·35. Found—Te, 22·02; Sb, 21·74; Cl, nil.

The action of antimony pentachloride on tellurium dioxide can be considered as first resulting in tellurium tetrachloride, which immediately unites with an additional molecule of antimony pentachloride, forming the addition product $TeCl_4SbCl_5$:—

(*a*) $5TeO_2 + 4SbCl_5 = 5TeCl_4 + 2Sb_2O_5$;

(*b*) $TeCl_4 + SbCl_5 = TeCl_4,SbCl_5$;

or—

$$5TeO_2 + 9SbCl_5 = 5[TeCl_4,SbCl_5] + 2Sb_2O_5.$$

The compound $TeCl_4.SbCl_5$ appears in white tabular crystals which are readily decomposed by water.

Phosphorus Oxychloride and Tellurium Dioxide.—When phosphorus oxychloride is brought in contact with tellurium dioxide and the two are allowed to remain together in a warm place, a crystalline mass of large flat monoclinic plates begins to form. The excess of phosphorus oxychloride can be readily removed by means of carbon bisulphide. These crystals are deliquescent, and are readily decomposed by water.

Analysis, $TeCl_4.POCl_3$.—Calculated—Te, 31·32; P, 7·61; Cl, 61·06. Found—Te, 30·22; P, 7·90; Cl, 59·13.

While phosphorus oxychloride doubtless first reacts with tellurium dioxide to form tellurium tetrachloride, the tetrachloride, as soon as produced, unites with one molecule of the excess of phosphorus oxychloride forming the addition product. The first reaction could be indicated $3TeO_2 + 4POCl_3 = 3TeCl_4 + 2P_2O_5$, and the entire reaction may be expressed $3TeO_2 + 7POCl_3 = 3[TeCl_4.POCl_3] + 2P_2O_5$.

When the above reaction is carried out with an excess of phosphorus oxychloride, a considerable portion of the reaction product remains in solution in the excess of the reagent.

It thus appears that by the action of the various anhydrous chlorides which have been studied on tellurium or tellurium dioxide, three series of products can form; either tellurium tetrachloride or the dichloride can be produced, or by the use of such compounds as phosphorus oxychloride or antimony pentachloride, double chlorides can be obtained.—*Journal of the American Chemical Society*, xxx., No. 5.

Neutral Triple Sulphates of Calcium.—J. D'Ans.—Solutions of copper sulphate give with potassium syngenite in salt solutions a new crystalline substance which is analogous to polyhalite $Ca_2MgK_2(SO_4)_2.2H_2O$, but contains copper instead of magnesium. The investigation of corresponding triple sulphates of calcium shows that the magnesium in the salts of formula $Me.SO_4.Me_2SO_4.6H_2O$ or $4H_2O$ can be replaced by copper, cadmium, nickel, cobalt, iron, manganese, or zinc, while the potassium can be replaced by ammonium, rubidium, or cæsium. Sodium does not give a triple sulphate with calcium. These new polyhalites crystallise very eadily.—*Berichte*, xli., No. 8.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

Introduction.

THIS article is a continuation of a preceding one (*Journ. Am. Chem. Soc.*, 1907, vol. xxix., p. 137 *et seq.*), in which were presented the first two parts of this system of qualitative analysis, dealing respectively with the preparation of the solution, and the analysis of the silver, copper, and tin groups (see CHEMICAL NEWS, vol. xciii., p. 134 *et seq.*; xcv., p. 89 *et seq.*). For the purposes of this investigation, for the general considerations underlying it, and for various conventional matters relating to its presentation, the reader is referred to the introduction to the preceding paper.

Although the final form of the scheme of analysis of the groups here considered has been worked out during the past year by the authors of this article, much of the preliminary experimental work, especially that relating to the rarer metals, was carried on by others in the Research Laboratory of Physical Chemistry of the Massachusetts Institute of Technology. It is, unfortunately, scarcely practicable to indicate in just what respects each of these investigators has contributed to the final result; but we wish to express in a general way our great indebtedness to Messrs. Howard I. Wood, Bart E. Schlesinger, and Charles Field (3rd) for the assistance which their work has been to us.

The present publication deals with the analysis of the precipitate produced by ammonium hydroxide and sulphide in the filtrate from the hydrogen sulphide precipitate. In addition to the seven common elements (nickel, cobalt, iron, manganese, zinc, chromium, and aluminium) considered in almost all schemes of qualitative analysis, we have included six of the especially important rarer elements, namely, beryllium, uranium, vanadium, thallium, titanium, and zirconium. The portions of the procedure and of the notes referring to these rarer elements are, however, marked with asterisks, so that they may be readily omitted by any one interested only in the common elements.

The general features of our scheme for the analysis of this solution will be most readily comprehended by an examination of the tabular outline presented in Tables VII. to IX. The considerations which have led to the adoption of this procedure will be discussed in the next chapter entitled "General Discussion," and the detailed process and the explanations of it will be presented in the following one entitled "Procedure and Notes." Later chapters, as in the preceding publication, will be devoted to the "Test Analyses and to Confirmatory Experiments and References."

General Discussion.

1. With respect to the original precipitation of these elements, schemes of qualitative analysis differ as to whether ammonium hydroxide and ammonium sulphide be used successively with a filtration between, or whether they be added together so that all the elements are contained in a single precipitate. The former of these methods has the serious disadvantage that the separation with ammonium hydroxide of the trivalent elements, aluminium, chromium, and ferric iron, from the bivalent elements, nickel, cobalt, manganese, and zinc, while satisfactory enough when certain combinations of these elements are present, is not so for other combinations. Thus even a large quantity of zinc may be quantitatively precipitated by ammonium hydroxide when a larger proportion of chromium is present; and manganese will in any case be partially precipitated by that reagent owing to its oxidation by the air to the manganic state, and it will be completely precipitated when phosphate is present in the solution. It is therefore necessary to provide for the detection of zinc and manganese both in the hydroxide and in the sulphide precipitate; and thus the scheme is complicated rather than simplified by precipitating separately with these reagents.

We have therefore adopted the plan of a single precipitation by simultaneous addition of both reagents, provision being made, however, for observing the effect of the addition of ammonium hydroxide alone for the sake of the indications which it may furnish. If care be taken to avoid an unnecessary excess of both the hydroxide and sulphide, all the elements in question are completely precipitated by these reagents even in moderately dilute solution with the exception of a little of the nickel and a variable proportion of the vanadium. The nickel can be removed from the filtrate by boiling. Even a large quantity of vanadium remains completely in solution when it is present alone; but, on the other hand, it may be almost completely precipitated, probably as a hypovanadate or vanadate, when certain other elements of these groups are present. Many experiments were made in this laboratory by Mr. Charles Field (3rd) to devise a practical method of reducing vanadium to a vanadous salt (corresponding to the oxide, V_2O_3) in which state it is completely precipitated by ammonium hydroxide. In accordance with the results of Gooch and Curtis (*Am. Journ. Sci.*, 1904, [4], xvii., 45) hydriodic acid (best in the form of a mixture of ammonium iodide and hydrochloric acid) was found to be the only available agent, but even with the aid of this reagent reduction and precipitation were never quite complete, and sometimes did not take place at all. For this reason it was not considered worth while to use this reagent, especially since the vanadium can be removed from the ammonium sulphide filtrate by acidifying, adding ferric chloride, and making alkaline with ammonia.

2. The ammonium sulphide precipitate in all the schemes of analysis known to us is first treated with cold dilute hydrochloric acid, in order to separate nickel and cobalt from the other elements. In spite of the general use of this process, we have become convinced that it does not fulfil the requirements of exact qualitative analysis; for not only is it true, as is generally known, that a considerable quantity of nickel and cobalt dissolves in a mixture of 1 volume of HCl (1·12) with 5 volumes of water when there is a large residue containing these elements, but our experiments have shown that a moderate quantity of either of them (up to at least 5 mgrms.) may *completely* dissolve, and thus escape detection when it was originally disseminated through a large precipitate of iron sulphide. We have therefore eliminated this treatment as a method of separation, and, after adding hydrochloric acid at first to decompose such of the sulphides as will act upon, and to get an indication as to the presence of much nickel or cobalt, nitric acid is added, so as to bring all the elements into a single solution. Incidentally it may be mentioned that our experiments support the view that the fact that nickel and cobalt sulphides, though not precipitated by hydrogen sulphide from a slightly acid solution, yet dissolve difficultly in a much stronger acid, is due to an abnormally slow rate of solution of these sulphides, which are, in fact, relatively soluble substances, at least in the freshly precipitated state. For these experiments have shown that nearly all of a portion of the precipitated nickel sulphide dissolves when treated with successive portions of cold dilute HCl (1 volume HCl, 1·12, with 5 volumes of water) even when the acid is kept saturated with hydrogen sulphide, and that solution continues to take place even after considerable nickel (30 mgrms. in 30 cc.) has passed into solution. When, therefore, the surface exposed to the acid is greatly increased, either by the residue being a large one or by a small residue being left in a finely divided state by the dissolving out of iron sulphide, a considerable quantity of nickel and cobalt passes into solution in a comparatively short time. It is interesting to note that the

* From the *Journal of the American Chemical Society*, xxx., No. 4.

reverse reaction, the precipitation of nickel sulphide by hydrogen sulphide in acid solution, also takes place very slowly, for Baubigny (*Comptes Rendus*, 1882, xciv., 963 *et seq.*; xcv., 35) has observed that in the presence of acetic acid, or of very small amounts of sulphuric or hydrochloric acids, the precipitation is a slow but continuous process.

3. Having now all the elements together in solution, the next step in our process is to divide them into two main groups by the addition of sodium hydroxide and peroxide, followed by subsequent boiling. This method has been previously applied by other authors to the separation of certain of the common elements, but not, we believe, as a general means of subdivision. These reagents cause the complete precipitation of iron, nickel, cobalt, and thallium, as hydroxides of the trivalent form, and of manganese, titanium, and zirconium as hydroxides of the quadrivalent form. We shall designate all these elements so precipitated as the "iron group." All the remaining elements, namely, aluminium, beryllium, zinc, chromium, uranium, and vanadium, remain in solution in the form of sodium salts of the corresponding acids, namely, as aluminate, zincate, chromate, peruranate, and vanadate. We shall designate all these elements so dissolved as the "aluminium group." The separation of the two groups by this process is entirely satisfactory, at any rate, from the standpoint of qualitative analysis, with the single exception that when only 5 or 10 mgrms. of zinc are present, this may be carried down completely when elements of the iron group (especially manganese) are present in large quantity. This makes it necessary to provide for the detection of zinc in the analysis of the precipitate when it is large, but this is not attended with special difficulty. The use of sodium peroxide has the distinct advantage over that of sodium hydroxide alone, that chromium, uranium, and vanadium are taken completely into solution, whereby not only a division of these elements between the precipitate and filtrate is avoided, but also the carrying down of zinc into the precipitate is made less common and less considerable.

4. Since, owing to the possible presence of phosphate, oxalate, or hypovanadate in the original solution, the alkaline-earth elements may be precipitated by ammonium hydroxide and sulphide, sodium carbonate is added with the hydroxide and peroxide, in order to ensure the complete precipitation of these elements (more especially barium) with the iron group. The presence of phosphate and carbonate does not affect at all the separation of the elements of the aluminium and iron groups from each other.

5. The separation of the elements of the aluminium group from each other is very simple when only the common elements, chromium, aluminium, and zinc, are to be provided for, and the process recommended below for this case offers no original features. It consists in precipitating the aluminium hydroxide from the solution with ammonium hydroxide after acidifying with nitric acid, the chromate in the filtrate with barium chloride after acidifying with acetic acid, and the zinc with hydrogen sulphide in the filtrate from the barium chromate.

(To be continued).

Isomeric Aminophenylarsenic Acid.—A. Bertheim.—When arsenic acid is heated with aniline *p*-aminophenylarsenic acid, $NH_2.C_6H_4.AsO(OH)_2$, is obtained. When phenylarsenic acid is nitrated a nitrophenylarsenic acid is formed which Ehrlich and Bertheim suggested should yield the isomeric meta-amino-acid on reduction. But reducing agents readily attack the arsenic acid residue. If, however, sulphuretted hydrogen is used and the sulphur is removed by a copper salt, the meta-aminophenylarsenic acid is obtained; or sodium amalgam, which does not affect the arsenic acid residue, can be used for the reduction. It is not quite certain whether the new acid (which differs in physical and chemical properties from the para acid) is the ortho or meta derivative, but it is most probably the latter.—*Berichte*, xli., No. 8.

UNIVERSITY OF CAMBRIDGE.

Honours for Men of Science.

On June 17th Honorary Degrees were conferred on the following men of eminence in Science, Literature, and the Arts:—The Right Hon. H. H. Asquith, The Duke of Northumberland, The Earl of Halsbury, Admiral Sir John Fisher, Sir Hubert von Herkomer, The Hon. C. A. Parsons, The Right Hon. Sir G. O. Trevelyan, Bart., Sir James H. Ramsay, Bart., Sir Andrew Noble, Bart., Sir William Crookes, Mr. Rudyard Kipling, Professor Horace Lamb, Professor G. D. Liveing, Mr. Alfred Marshall.

Dr. Sandys, the Public Orator, spoke as follows in presenting to the Chancellor the two chemists who were among the recipients of the Honorary Degree of *Doctor Scientiæ*. "Dignissime domine, domine Cancellarie, et tota Academia:—

"Cancellarii nostri auspiciis nunc demum eum ipsum praesentem videmus quem diu desideravimus, quem alia ex alia scientiarum in studiis occupata Societas praesidem suum olim suspexit. De scientia chemica et physica exploranda per annos plus quam quinquaginta praeclare meritus, in rerum primordiis primis spectri (ut aiunt) auxilio examinandis eam ipsam provinciam feliciter exploravit, quae etiam scientiae physicae in officina nostra maximo cum fructu indagata est. In observando quam acutus est, in experiendo quam peritus, in rebus difficillimis investigandis quam pertinax! Viri tanti exemplo Vergili dictum denuo praeclare confirmatum est:—'Labor omnia vicit improbus.'

"Duco ad vos Regiae Societatis socium, virum numismate Copleyano honoris causa haud ita pridem donatum, equitem insignem, Willelmum Crookes."

"Abhinc annos duo et sexaginta Cantabrigiam primum petivit vir intra proximum quinquennium in disciplina mathematica et in rerum naturae studiis honores summos adeptus, qui, post itinera sua Berolinensia, scientiae chemicae inter nosmet ipsos docendae sese strenue dedicavit, et Divi Ioannis in Collegio primam scientiae illius officinam Cantabrigiensem aedificandam curavit. Postea Universitatis professor nominatus, horum studiorum officinae amplissimae publice condendae summan diligentiam, summam operum exterorum experientiam, feliciter adhibuit. Lucis vero radiis spectri (ut aiunt) ope retexendis, et rerum naturae penetralibus examinandis, quot annorum labores dedicavit! quot discipulis studiorum uno tenore assidue peractorum exemplar quam pulchrum praebuit! In operibus bonis adiuvandis liberalissimus, in negotiis academicis partium liberalium defensor indefessus, per tot annos inter tantas rerum vicissitudines animum serenum, aequum, prudentum, modestum conservavit. Virum talem preconio eodem dignum esse crediderim, quo populi Americani praeses quidam magnus, professoris nostri in anno primo munus suum ingressus, populi sui a poeta eximio postea est laudatus:—

> En vir benigne intentus, fortis, providus,
> Sagax patiensque, laudem non culpam timens.

"In laboribus suis ad exitum felicem perducendis per annos plurimos supersit Collegii sui socius perpetuus, scientiae chemicae professor emeritus, Georgius Downing Liveing."

(*Translation*).

Sir William Crookes.

Under the auspices of our Chancellor we now have before us one we have long desired to see, a man whom so many scientific Societies have claimed as their President. For more than fifty years he has greatly distinguished himself in chemical and physical research. The investigation of the spectra of the so-called primordial elements he has made his own special province—a province also which has been explored not unsuccessfully in our own laboratories of physical science. As an observer how acute he is! As an experimentalist how skilful! In overcoming diffi-

culties how persistent! In such as he we see once more signally confirmed the well-known statement of Virgil:—"Labor omnia vicit improbus." (All difficulties yield to strenuous toil).

I present to you a Fellow of the Royal Society, a recent Copley Medallist *honoris causa*, the worthy Knight, WILLIAM CROOKES.

PROFESSOR G. D. LIVEING.

Sixty-two years ago he first entered Cambridge; in the next five years he obtained the highest honours in Mathematics and Physical Science. After a residence in Berlin he earnestly devoted himself among us to the teaching of chemistry; at St. John's he superintended the building of the first laboratory for that science. Subsequently appointed Tutor in the University, he applied with success his great experience in other fields of labour, his great diligence in building a very complete public laboratory for this science. In revealing the secrets of the spectrum and in investigating the problems of Nature, what years of toil did he spend! and to how many of his scholars did he afford at the same time an excellent example of much work assiduously performed. Most liberal in assisting good work, in the business of the University he was an untiring champion. During all this time, in all these vicissitudes he preserved a calm demeanour, an evenly balanced mind, and exhibited the greatest modesty. He is worthy of the same compliment as a certain President of the United States who entered upon his office in our Professor's first year: he received the following laudatory notice from the poet Lowell:—

"Behold a man, kind, energetic, brave,
Farsighted, patient, fearing praise more than blame."

In his labours, carried out to his resignation, for many years he has been a life-member of our College, a worthy Professor of Chemistry, GEORGE DOWNING LIVEING.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Special General Meeting, June 12th, 1908.

Dr. CHARLES CHREE, F.R.S., President, in the Chair.

A SPECIAL General Meeting was held at which the resolution passed at the Special General meeting on May 22nd was confirmed.

An Ordinary Meeting of the Society was then held.

A paper by Messrs. E. BELLINI and A. TOSI on "*Experiments on a Directive System of Wireless Telegraphy*" was read by Mr. Tosi.

The authors describe the results obtained in the course of their work upon a further development of their original directive system.

In the earlier method previously described (*Electrical Engineering*, 1907, ii., 771; 1908, iii., 348) it was not possible to say from which side of the receiving station the transmitted waves arrived, for though the radiation was practically confined to the plane of the aërial system, it was emitted equally in the opposite direction to that desired.

In the new unilateral system the waves are sent in a single direction only, and the problem of getting rid of the backwardly extending radiation has thus been solved. The method adopted consists in superposing a bilateral directive system, as previously described, upon an ordinary or vertical antenna system. Since the two half-diagrams of the directive aërial are opposed in phase, it follows that when the two systems (directive and vertical antenna) are simultaneously excited and in phase, the one half of the directed radiation will add itself to, and the other subtract itself from, the radiation due to the vertical antenna system. The diagram of the vertical antenna system being a circle, the resultant diagram of the superposed systems will, for the case where these are in phase as regards excitation, be a cardioid whose maximum radius vector is double that of the diagram of the directive system alone. Since then the directive system is able to vary the direction of its maximum emission by means of the radiogoniometer, it follows that by moving the position of this latter it is possible to shift the direction of the resultant emission represented by the cardioid, in a corresponding manner. Several energy diagrams obtained by means of the thermo-galvanometer are reproduced in the paper, and these show that, even where the excitations are not exactly in phase, the only result is a slightly less good diagram. Diagrams of the scheme of connections employed during the taking of the energy diagrams are also given.

The same principle of the superposition of the two systems has been applied to the case of the reception. In this case a phase displacement of 90° in the excitations is produced in a suitable manner, and in this way, when the pointer of the radiogoniometer is directed, say, towards the transmitting station, the reception is a maximum, whilst when turned at 180° from this it is a minimum, or zero. Diagrams of received energy and of the connections employed are also given in this case.

The system of unilateral directive wireless telegraphy described in the present paper is of special interest owing to the facility with which it is possible to change over from one system to the other; thus, from the ordinary vertical antenna system to the bilateral directive or the unilateral directive, or *vice versâ*. The aërial arrangements, moreover, remain exceedingly simple.

When a message from a station of unknown position is expected, the vertical antenna or ordinary system would be employed; on once effecting reception, one can pass to the bilateral or unilateral directive system, and thus determine the direction and on which side the transmitting station lies, at the same time making oneself independent of other transmissions.

In the same way, with the transmission, the vertical antenna would be employed for calling up an unknown station, or for simultaneously sending to several stations; on once getting a reply the operator can then readily determine the position of the receiving station, with the aid of the unilateral system, and thenceforth will transmit solely in that direction.

Attention is called to the advantages which such a directive system offers in the case of the commercial services as well as for military and naval purposes.

Dr. FLEMING said that he had long been acquainted with the interesting experiments of the authors of this paper, and desired to express his congratulations on the skill and inventiveness with which these investigations had been prosecuted. He was glad to see that the experiments of the authors confirmed in many ways the theory which he (Dr. Fleming) had given of the operation of a bent antenna as employed by Mr. Marconi.

When Mr. Marconi read his paper at the Royal Society in March, 1906, describing his experiments on directive telegraphy he gave no theory in the matter, but Dr. Larmor pointed out in the discussion that an antenna partly vertical and partly horizontal was equivalent to the sum of a magnetic oscillator and an electric oscillator, and shortly afterwards he (Dr. Fleming) had gone more carefully into the matter mathematically, and showed that the observed effects could be accounted for on this theory.

Both Mr. Marconi and he himself had obtained by the same methods as those employed by Messrs. Bellini and Tosi the same type of pear-shaped radiation curves obtained by the authors of the paper by combining together the effect of closed and open oscillators. Although this theory had been criticised by Dr. Mandelstam lately, yet nevertheless there did not seem sufficient grounds for objecting to it. Mr. Marconi had, as everyone knew, employed directive antennæ for a long time past in his power stations at

Poldhu and Clifden, and had also given demonstrations showing that the position of ships out of sight could be located by means of such receiving directive antennæ. Nevertheless, Messrs. Bellini and Tosi had worked out extremely ingenious arrangements for determining the direction of the radiant point without moving the antennæ themselves. He (Dr. Fleming) had also shown that, having the power to locate the radiant point, two stations equipped with such antennæ at a known distance apart could by simultaneous observations determine also the distance of the radiant point, and this might become important in connection with marine work.

It ought also to be noticed that Dr. F. Braun, of Strasburg, had by the employment of three open antennæ having oscillations in them of definite phase difference, been able to obtain radiation curves having the form of a cardioid similar to some of those given by the authors of the paper.

Dr. W. H. ECCLES warmly congratulated the authors upon their beautiful and original method. On this method, by merely rotating a small coil of wire on the table, a fixed aërial directive system of any size was made to do what could otherwise only be done by turning the whole system of aërial wires in azimuth. The essence of the system was the piece of apparatus styled the radiogoniometer, which, by causing appropriate component radiation from two fixed wire triangles set at right angles, brought about a resultant radiation in any direction desired—just as if a virtual aërial of the full size of the fixed aërials were being rotated in the air. Someone had compared the result so achieved with that obtained by Marconi's well-known arrangement of a number of fixed bent antennæ with their horizontal portions directed from a centre to various points of the compass. Apart from the fact that it is rarely feasible to fix up a large number of antennæ, the radiogoniometer method has the advantage that the direction of transmission or reception can be altered perfectly smoothly; so that if it can be used, as stated by the authors, accurately to about 1° of arc, the radiogoniometer is the practical equivalent of 360 bent antennæ.

The looped aërials used by the authors did not seem to the speaker to be the best kind of radiator for utilising the principle they had developed. The radiogoniometer would prove to be capable of giving excellent results with two fixed antennæ of the bent type set at right angles, one, for example, in the meridian plane and the other in the east-west plane. The two looped aërials of the authors may each of them be assimilated to a pair of vertical antennæ emitting waves of 180° phase difference, and with the assumption that for a single vertical aërial the inverse square law holds for the propagation of electrical effects from a single aërial, the speaker showed by aid of the ordinary equation of wave-motion that a looped antenna obeying the condition stated emitted two waves of equal period, of phases differing 90° and 180° respectively from the phase of the radiation from either side of the loop, and of amplitudes having a ratio proportional to λ/x. This means that one portion of the resultant radiation obeys an inverse square law and the other portion an inverse cube law; the latter portion is the more important of the two when the distance x is small, the former is the more important when the distance x is much greater than the wave-length λ. This explains why Dr. Fleming's measurements of the radiation from closed oscillators, which were carried out at short distances, gave small promise of powerful propagation to a distance, and why Messrs. Bellini and Tosi had found as a fact that good propagation occurred to great distances.

As regards the phases of the two portions of the radiation, the important portion for wireless telegraphy, namely, that obeying the inverse square law, was 90° out of phase with the near side of the loop; and thus it is clear that the vertical aërial the authors had used to cancel the radiation to one side of the sending station, must for that purpose emit radiation 90° out of phase with the near side of the loop. Messrs. Bellini and Tosi had reached this conclusion in the course of their experiments.

After eliciting that the power-supply to the primary of the induction-coil for the Dieppe-Havre experiments was 500 watts, Dr. Eccles concluded by remarking that the authors were worthy of special congratulation because in their method of unilateral transmission by phase and intensity adjustment of several radiators they had eluded the difficulties of managing the phases that had apparently baffled F. Braun when he was using the same principle.

Mr. W. DUDDELL complimented the authors upon their paper, and asked for the wave-length of the waves given out by the transmitter. He also asked what would be the effect of opening the closed antennæ at the top.

Mr. TOSI, in reply to Dr. Fleming, stated that Braun's cardioid diagram was a theoretical diagram only; the method had not succeeded, and no actual diagram obtained experimentally had ever been shown.

Referring to the similarity which Dr. Fleming said the pear-shaped diagram of Marconi bore to the authors' diagrams, Mr. Tosi could only say that they had purposely shown some bad diagrams as well as some good ones. The good diagram of Marconi's corresponded to the authors' bad diagram. With the Marconi horizontal aërial it would, moreover, be necessary, as Dr. Eccles had pointed out, to employ 360 such wires to obtain an accuracy in locating the bearing of the radiant point equal to that secured with the system now described. Further, if the emitted energy is actually represented by Marconi's pear-shaped diagram, it is difficult to understand why the transatlantic messages from Clifden to Glace Bay are readily picked up off the Algerian coast, at right angles to the line of transmission.

In reply to Mr. Duddell he said that the wave-length of the waves employed was from 350 to 400 metres.

A paper "*On the Lateral Vibration and Deflection of Clamped-directed Bars*" was read by Dr. J. MORROW.

This is an investigation of the problems which arise in connection with the lateral vibrations of clamped-directed bars. The term "directed" is used to describe the extremity of a bar which is constrained to maintain its original direction, but is free to take up any position of lateral deflection. These terminal conditions are mentioned in Rayleigh's "Sound," but are dismissed on the ground that the directed end cannot be realised experimentally. In the present paper, however, it is shown that the "directed" end is of great importance and of frequent occurrence in engineering practice; and, further, that by the aid of a simple device, it can easily be investigated in the laboratory.

The first problem is the ordinary one of a bar vibrating under the effect of its mass alone. The frequencies of the fundamental and harmonics are determined. The equation of the curve assumed at any instant by the elastic central line is obtained, and the positions of the nodes found.

When the bar vibrates under the influence of a longitudinal force in addition to its own mass, the problem is more difficult. The solution is exact up to a certain point, after which it is assumed that the longitudinal force is confined within certain limits. An approximate solution is then obtained which should be satisfactory for nearly all purposes.

Other portions of the paper deal with further cases of loaded and unloaded bars, both with and without longitudinal force. In each problem means are supplied by which the necessary magnitude of the constraining couple at the directed end can be found.

When the mass of a bar, in addition to that of a concentrated load, is taken into account, the method used is that of continuous approximation previously developed by the author. The method is found to be as readily applicable to this as to other cases.

In the last section an account is given of a few experiments on clamped-directed bars, and the results are seen to be in complete agreement with the theory. The paper

also contains the solutions of some cases of deflection under static loads.

A paper entitled "*On the Resistance of a Conductor of Uniform Thickness whose Breadth suddenly changes, and on the Shape of the Stream-lines*," was read by Prof. C. H. Lees.

A knowledge of the resistance of a conductor whose section suddenly changes is of considerable practical importance, but mathematical difficulties have prevented an exact solution of the problem. Lord Rayleigh has given an approximate solution of the case in which a cylinder of circular section is joined at one end to the plane surface of a large conducting solid; and Prof. Hicks has solved the case of a wire of small diameter ending in the surface of a conducting sphere. The mathematical difficulties of the problem disappear if the conductors are of rectangular section and one dimension; *e.g.*, the thickness remains constant, while the breadth suddenly changes and the two are joined together either with their axes or with two sides collinear. The paper shows that the resistance between two transverse sections through points situated at considerable distance from the change of section on opposite sides of it is equal to the sum of the resistances of the portions of conductor between each of the two sections and the change of section, each considered as part of an infinite length, plus the resistance of a length of either conductor equal to its breadth multiplied by an expression given.

Dr. Russell congratulated the author on having obtained the exact solution of an important problem, and thanked him for giving it in a form in which it could be utilised readily by electricians. Somewhat similar problems are of frequent occurrence in practice; in particular he instanced the measurement of the resistance of the bonds connecting the rails in electric tramway systems. The difficulty in this case is in knowing where the rail ends and the bond begins.

A small variation in the position of the potential contacts makes a large variation in the reading of the galvanometer. In many cases the only way of attacking the problem is to calculate the resistance from the known resistivity of the metals by the approximate method indicated by Maxwell. An exact solution, therefore, like the one obtained by the author, would be of great value in checking the accuracy of the approximate method.

A paper on "*The Inductance of Two Parallel Wires*" was read by Dr. J. W. Nicholson.

When direct and return currents flow in two wires of great length, and the alternation is not rapid, the effective self-induction per unit length of the system may be calculated readily by simple integration. If the wires have radii a, b, and permeabilities μ, ν, and if C be the distance between their axes,—

$$L = 2 \log c^2/ab + \tfrac{1}{4}(\mu+\nu).$$

This formula is often of little practical use when the frequency of alternation is several thousands per second. Such frequencies are of constant use in practical work. For example, in the measurement of small inductances by Mr. Campbell's method it is necessary to employ long leads in order to keep them at some considerable distance from the bridge and other circuits. The self-induction of these leads must be small, and a calculation of its value is very desirable. The general case presents very great mathematical difficulty, but the solutions given in the paper appear to include most cases of practical utility. The mode of proof depends upon a transformation of a harmonic series containing the Bessel functions of type k_n finite at infinity to a similar series with respect to a new origin.

Mr. A. Campbell congratulated the author, and said it was often desirable when measuring self-inductances to check the results by calculation. The author's formulæ would be useful in finding the inductance of parallel leads, although this was best obtained experimentally as a separate experiment.

Dr. Russell said that it required considerable mathematical skill and great patience to attack this problem, and so he thanked the author for having obtained a solution sufficiently accurate for practical purposes. He pointed out that the formula would have to be used with caution at very high frequencies as the capacity current was then appreciable. He suggested to the author that he should compute the kinetic energy of the ions or electrified particles by the motion of which modern theory explains the phenomena of the electric current. It has hitherto been the custom to assume that the self-inductance of a current flowing in a circuit can be calculated by finding the magnetic energy only, and this is what Dr. Nicholson has done. If we accept modern theory, however, we must admit that part of the total energy of a current is due to the kinetic energy of the ions. To take this into account it is necessary to add the term $\Sigma m v^2/i^2$ to Dr. Nicholson's solutions, where m is the mass of an ion, v the mean velocity given to it by the electromotive force, and i the current. In the case of metallic conduction this term is in general negligible compared with those found by the author. In cases, however, where the current takes place through gases—as in vacuum tube lighting—the value of the correcting term is appreciable, owing to the high velocity of the electrified particles. In a paper to the Röntgen Society on the measurement of a current through an X-ray tube, Mr. Duddell recently described some very interesting phenomena which he had noticed during his difficult but very successful experiments. The speaker thought that probably some of these effects might be explained by taking into account the kinetic energy of the moving ions.

A paper by Dr. Barkla and Mr. Sadler on "*Homogeneous Secondary Radiation*" was read in abstract by the Secretary.

Two papers by Prof. Morton entitled "Note on the Amount of Water in a Cloud formed by Expansion of Moist Air," and "An Elementary Treatment of the Motion of a Charged Particle in a combined Electric and Magnetic Field," were taken as read.

NOTICES OF BOOKS.

The Patents and Designs Act, 1907. By James Roberts, Esq., and H. Fletcher Moulton, Esq. London: Butterworth and Co. 1907.

The object of this exposition of the Patents and Designs Act of 1907 is to help the reader to interpret the provisions of the new Act and to forecast its probable effects. The difficulties which are likely to arise owing to ambiguity or obscurity of wording are pointed out, and the references to typical cases and decisions throw light upon the working of the Act. In the introduction the most important changes in the law are summarised.

Développement et Progrès de la Fabrication du Malt pendant les Quarante Dernières Années. ("The Development and Progress of the Manufacture of Malt during the last Forty Years"). By Ed. Eckenstein. Paris: A. Hermann. 1908.

The evolution of the more scientific and labour-saving processes which have replaced or are replacing the primitive rule-of-thumb methods originally employed in malting is described in a somewhat partial spirit in this book. Continental methods are given much greater prominence than those adopted in England, and certain reforms and workers are picked out for special mention, perhaps above their deserts, while other investigators whose researches have been at least of equal value are neglected. However, the history of the development of pneumatic and mechanical malting is fully discussed, and as the author has had much varied experience in constructing modern machinery, he brings an intimate practical knowledge to bear upon his subject. The book includes a bibliography of works on brewing and malting, practically only French

and French translations of German books being comprised in the list. In addition a short review is given of questions concerning malt and malting which have been discussed in the *Österreichische Zeitschrift für Bierbrauerei* during the period of which the book treats.

Nitrate Facts and Figures, 1908. By A. F. BRODIE JAMES, F.S.S. London: Fredc. C. Mathieson and Sons.

THE annual publication of abstracts of the balance sheets of the principal Nitrate and Railway Companies gives the investing public some opportunity of acquiring knowledge which might otherwise be difficult to obtain. Statistics relating to capital, dividend, reserve, output, &c., are tabulated in these notes, and the routes of the nitrate railways with their proposed extensions are shown in three maps. In the preface the author calls attention to the risks which shareholders run if they do not do their utmost to obtain regular monthly advices concerning production and other details of the running of the company in which they are interested.

The Fundamental Conceptions of Chemistry. By Dr. S. M. JÖRGENSEN. Translated by M. P. APPLEBY, B.A. London and Brighton: Society for Promoting Christian Knowledge. New York: E. S. Gorham. 1908.

A BOOK of this small size with what appears to be a somewhat pretentious title at once arouses in the mind the questions whether it is likely to find readers who will benefit by the study of it, and whether it can be recommended to the student as better worth his perusal than selections from one of the many excellent treatises on chemistry which have gained a world-wide reputation, and which are to be obtained in all languages. On the whole these questions may be answered in the affirmative. The book deals rather with general ideas than with details, and the conceptions of physical chemistry are introduced early and are well explained. Experiments which illustrate fundamental principles, and which require only comparatively simple reagents and apparatus, are fully described, and the historical growth of the science is emphasised, every advance being connected with the name of some great chemist. The book may safely be put into the hands of beginners whose instruction wants supplementing, and they will find it both lucid and interesting to read.

Alkali Soils, Their Nature and Reclamation. By FRANK T. SHUTT, M.A. Ottawa: Department of Agriculture.

IN this Bulletin an account is given of the composition and characteristics of white and black alkali incrustations upon the soils of countries in which the rainfall is deficient, and different methods of removing, or lessening the amounts of, the injurious constituents are explained. The subject is one which has more interest for the farmers of the North-West plains of Canada and British Columbia than for English agriculturists, to some of whom, however, an accumulation of saline matter in hot weather upon land with a sandy, porous subsoil is not unknown.

CORRESPONDENCE.

THE POTENTIAL ENERGY OF THE ELEMENTS.

To the Editor of the Chemical News.

SIR,—I have read with interest Mr. Daniel J. Rankin's paper on the above subject in the CHEMICAL NEWS for June 26th.

It is, however, not apparent from the paper how the values for the "potential energy of the elements" given in the table were originally calculated; I should be obliged if the author would kindly enlighten me upon this matter.—I am, &c.,

H. STANLEY REDGROVE.

The Polytechnic, Regent Street, W.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Berichte der Deutschen Chemischen Gesellschaft. Vol. xli., No. 8, 1908.

Action of Chlormethylsulphate on Amino Groups.—J. Houben and Hans R. Arnold.—Methyl sulphate acts as a methylating agent on aminobenzoic acid, attaching itself to the amino group, and methyl sulphuric acid salts of methylamino acids are formed:—

$$C_6H_4(COOH).NH_2 + SO_4(CH_3)_2 = C_6H_4(COOH).NH.CH_3SO_4H.CH_3.$$

With chlormethylate sulphate the methyl sulphuric acid salt of a chlormethylamino acid might result, or the chlormethyl sulphuric acid salt of a methylamino acid:—

$$C_6H_4(COOH).NH_2 < \begin{matrix} CH_2Cl \\ SO_4.CH_3 \end{matrix},$$

$$C_6H_4(COOH).NH_2 < \begin{matrix} CH_3 \\ SO_4.CH_2Cl \end{matrix},$$

With water chlormethylsulphate gives methyl but not chlormethylsulphuric acid. Thus the chlormethyl group is probably more reactive than the methyl group. When chlormethylsulphate acts on the amino group the chlormethyl group is split off and introduced into the amino residue. Thus when anthranilic acid and chlormethylsulphate react in ethereal solution, a white precipitate, which possibly contains the salt of a chlormethylanthranilic acid, is formed. On warming hydrochloric acid is evolved, and the salt of methylene anthranilic acid is obtained. The free acid polymerises very rapidly. Methylanthranilic acid and chlormethylsulphate should yield chlordimethylanthranilic acid, but again polymerisation takes place, methylene-bis-methyldianthranilic acid being formed—$C_6H_4(COOH).N(CH_3).CH_2.(CH_3)N.C_6H_4.COOH$. Ammonia reacts with chlormethylsulphate, giving the hydrochloric acid salt of methylamine, ammonium sulphate, and hexamethylenetetramine. Amines react differently; thus diethylamine yields tetraethylmethylenediamine—

$$2(C_2H_5)_2NH + SO_4(CH_3)(CH_2Cl) = (C_2H_5)_2N.CH_2.N(C_2H_5)_2HCl + HSO_4.CH_3.$$

Action of Ammonia upon Phosphorus. Different Modifications of Phosphorus.—Alfred Stock.—Four varieties of phosphorus are known—the colourless, Schrötter's red variety, Hittorf's and Schenck's (light red). A black and a white modification have also been described. The last is undoubtedly not pure phosphorus, but contains water. The statements about black phosphorus are contradictory, and no definite conclusions can be drawn from them. However, Schenck's and the colourless variety undoubtedly darken in presence of ammonia. When liquid ammonia is allowed to stand with phosphorus at the ordinary temperature a slow reaction takes place. The liquid reddens and the phosphorus darkens, while a black mass is deposited. When warmed the red liquid and the black substance are formed more quickly, and above 85° the liquid becomes colourless. At 90° to 100° the molten phosphorus is changed into a black mass which has no further action upon ammonia, although it consists chiefly of unchanged phosphorus. The residue on evaporating off the ammonia was treated with carbon disulphide and filtered. It amounted to 25 to 75 per cent of the phosphorus used. It contained 74 to 81 per cent of phosphorus, 2 to 3 per cent of nitrogen, 1 per cent of hydrogen. By treatment of the black substance with aqueous acids a red substance was obtained which had approximately the composition of the suboxide P_4O. Probably, however, the so-called suboxide is not a single substance, and it appears to contain always 1 per cent of hydrogen. The red substance

when heated to 470° yielded a residue which consisted of phosphorus nitride. It reacted in the cold with ammonia, turning black. The chief effect of the action of ammonia upon phosphorus appears to be the conversion of the colourless modification into a red variety which is identical with Schenck's. Thus, exclusive of colourless phosphorus, the author believes that four modifications exist, and he thinks that certain elements can occur as isomeric molecules.

Compounds of Mercury Cyanide with Alkalis and with Nitric Acid.—K. A. Hofmann and H. Wagner.—Mercury cyanide readily forms crystalline compounds with caustic potash. From determinations of the freezing-point, conductivity, and saponification it is evident that the complex ions $HgC_2N_2.\overline{O}H$ are formed, and when the concentration is sufficient they give solid salts $HgC_2N_2.HOK + H_2O$, $HgC_2N_2.HONa + H_2O$. From a solution of mercury cyanide in strong nitric acid a crystalline product, $[Hg(CN)_2]_2NO_3H$, can be obtained. It explodes on heating, and the aqueous solution has a strong acid reaction.

Preparation of Benzilic Acid.—Hans v. Liebig.—Large quantities of benzilic acid free from benzoic acid can be prepared by dissolving equal weights of benzile and caustic potash in twice the weight of water, and adding alcohol. The vessel is then heated on the water-bath to boiling for ten to twelve minutes, and the contents are turned into a beaker. The crystalline mass is drawn off after some hours, dissolved in cold water, and dilute sulphuric acid is added to the filtered solution, when benzilic acid separates out.

Action of Alkalis on Ethyl Sodium Thiosulphate.—A. Gutmann.—Alcoholic potash gives with ethyl sodium thiosulphate a white crystalline precipitate of sulphite and an alcoholic liquid, which smells strongly of mercaptan when water is added. Possibly the reaction occurs according to the equation $SO_2(ONa).SC_2H_5 + KOH = SO_3NaK + (C_2H_5)SOH$, and the substance $(C_2H_5)SOH$ is a thioethylhydroperoxide. The oxygen in it is very loosely bound, and it may have the formula $C_2H_5.\overset{II.}{S}.O.H$ or $C_2H_5.\overset{IV.}{S}{:}O.H$ corresponding to ethylhydro-peroxide $C_2H_5.O{:}O.H$ or $C_2H_5.\overset{IV.}{\underset{\ddot{O}}{S}}.H$ corresponding to the sulphoxides $C_2H_5.\overset{IV.}{\underset{\ddot{O}}{S}}.C_2H_5$.

Oxidation of Brucine and Strychnine.—Hermann Leuchs. — Brucine and strychnine can be oxidised by potassium permanganate in acetone solution, and the products are crystalline acids. By cautious oxidation from both alkaloids a much smaller yield of a second alkaloid can be obtained. The formula of the first acid from brucine, brucinonic acid, is $C_{23}H_{24}O_8N_2$, and that of the acid from strychnine, strychninonic acid, is $C_{21}H_{20}O_6N_2$. Thus they differ from the corresponding alkaloid by an additional O_4, while H_2 less is present. The oxygen forms two carboxyl groups, possibly causing the breakdown of a carbon double bond, $\overset{H}{\underset{}{-C}}=\overset{H}{\underset{}{C-}}$. One of the carboxyl groups in the acid is neutralised by the basic nitrogen, while the other gives the compound its strongly acid character. The acids thus yield two kinds of esters —neutral mono-esters and basic diesters. As regards the hydrogen which is removed, probably a $>C<^{H}_{OH}$ group is transformed into a keto group. It is already known that in brucine and strychnine one oxygen atom is present in the form of acid amide group, $>N-CO$, and two more atoms in brucine form part of methoxy groups. The remaining atom is probably present in a secondary alcohol group, and thus the acids formed by oxidation must be ketone acids. The second acids produced by the oxidation are probably dihydro acids of formulæ $C_{23}H_{26}O_8N_2$ and $C_{21}H_{22}O_6N_2$ respectively. They contain two carboxyls and an alcohol group instead of a ketone group.

MISCELLANEOUS.

Geological Survey on the Coals of South Wales.—The Board of Agriculture and Fisheries desire to give notice of the publication of a Memoir of the Geological Survey on the Coals of South Wales. This Memoir gives the results of an investigation into the characters of the coals of South Wales, which was commenced in 1901. It includes not only the analyses which were made for the purposes of the investigation, but all previous analyses of recognisable seams, on which reliance can be placed, the total number amounting to 203. The sequence of the seams and their correlation throughout the coal-field is given, and the methods used in analysis of coal and the accuracy of coal analyses are discussed. The variations in seams, and in different parts of seams, are shown by a table, and the various classifications of coals are given. The volume is accompanied by ten plates illustrative of the sequence of seams, the distribution of different classes of coal, &c., several plates giving the positions of iso-anthracitic lines throughout the coal-field for certain seams or groups of seams. The anthracitic quality of the coals in a part of the coal-field is shown to have been in the main an original character and not the result of alteration. Copies may be obtained from any agents for the sale of Ordnance Survey maps, or directly or through any bookseller from the Ordnance Survey Office, Southampton, price 1s. 6d.

Royal Society of Arts.—The Annual General Meeting of the Royal Society of Arts was held on the 24th ult., Sir Steuart Colvin Bayley, K.C.S.I., C.I.E., Chairman of Council, in the chair. The business of the meeting was the reading the Report of the Council on the work of the Society during the past Session, the 154th since the formation of the Society in 1754. The Report, after a reference to the grant by His Majesty of permission to add the term Royal to the Society's title, gave a summarised account of the various papers read and lectures delivered during the past year. In referring to the award of the Albert Medal to Sir James Dewar for his Low Temperature investigations, special stress was laid on the power placed at the disposal of those interested in industrial applications of Science by the provision of temperatures so far below any hitherto available, and it was suggested that before long this new power would find numerous practical applications. Amongst the lectures delivered under the various Trusts available for such purposes, the course of lectures on Industrial Hygiene, provided out of funds left some years ago to the Society by Mr. Benjamin Shaw, was one of the most important. The award of a Medal, under the same Trust, to Professor W. Galloway, for his researches into the action of Coal Dust in Colliery Explosions, was also recorded. The number of papers worked at the examinations of the Society, just concluded, was well over 25,000, the highest number yet reached. The election of His Majesty the German Emperor, on the occasion of his visit to the country last year, as an Honorary Royal Member, was mentioned, as was also the death of two other Honorary Royal Members of the Society—the King of Sweden and the King of Portugal—during the year. At the conclusion of the reading of the Report, the result of the ballot for the election of the new Council was announced. His Royal Highness the Prince of Wales continues to hold the office of President, which he has filled since the accession of His Majesty in 1901.

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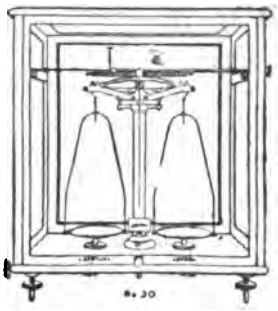

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ACTION OF SELENIUM AND TELLURIUM ON ARSINE AND STIBINE.*

By FRANCIS JONES, M.Sc., F.R.S.E.

IN a paper on stibine, read before the Chemical Society and published in the *Journ. Chem. Soc.* for 1876, p. 641, I pointed out that sulphur decomposes the gas in presence of light, with formation of hydrogen sulphide and antimony trisulphide which deposits on the sulphur. Further, that the liberated hydrogen sulphide also reacts with stibine, producing antimony trisulphide and free hydrogen : — (*a*) $2SbH_3 + 6S = Sb_2S_3 + 3H_2S$; (*b*) $2SbH_3 + 3H_2S = Sb_2S_3 + 12H$. It was shown that the reaction serves as an extremely delicate test for stibine, and it was also used to determine its composition, by estimating the amount of antimony deposited as sulphide on the sulphur, as compared with the amount of hydrogen in the hydrogen sulphide evolved. The value of this method for determining the composition of certain hydrides has since been shown by its application to the case of germanium hydride, which is also decomposed by sulphur (Uber Germaniumwasserstoff, von E. Voegelen, *Zeit. Anorg. Chemie*, xxx., p. 325).

I also showed that sulphur acts similarly on phosphine and arsine, but not so readily as in the case of stibine.

It appeared probable that the elements selenium and tellurium, which are analogous to sulphur, would act in a similar way with arsine and stibine, and I have now to record the results of experiments made to decide this question.

In the course of these experiments it was necessary to have distinguishing tests for the hydrides of sulphur, selenium, and tellurium, which are so alike in their reactions. Each of these hydrides produces on paper moistened with solution of lead acetate, dark stains which are extremely similar, and I was unable to find any tests recorded which would serve to distinguish them. On trying the effect of various reagents upon these stains I found that hydrogen dioxide and hydrochloric acid were useful for this purpose, at all events in the case of a distinct stain. The differences in the behaviour of these stains with reagents are shown in the following table :—

Stain.	H_2O_2.	HCl (dilute).	HCl (strong).
Lead sulphide	Stain rapidly fades and leaves paper white.	Stain fades almost instantly and leaves paper white.	Stain instantly fades and leaves paper white.
Lead selenide	Stain slowly fades and leaves orange coloured stain (selenium).	Stain slowly fades and leaves orange coloured stain	Stain slowly fades and leaves orange coloured stain.
Lead telluride	Stain fades in course of time.	Stain fades only slightly and leaves greyish black stain.	Stain fades only slightly and leaves greyish black stain.

These reactions were employed to discriminate between the lead stains obtained in the course of the experiments, except in those in which arsine was used, as this gas acts directly on solutions of lead and produces stains on paper moistened with lead acetate very similar to the others. This reaction will be referred to subsequently.

Action of Selenium on Arsine.

(*a*) *In Sunlight.*—The arsine employed was generated in the way described in the paper already referred to; that is to say, a solution of arsenious acid in dilute hydrochloric acid was allowed to drop from a stoppered funnel tube into a gas-bottle filled with pure ganulated zinc. The exit-tube from the gas-bottle passed to a wash-bottle containing dilute soda, and the exit-tube from this passed to a drying tube filled with calcium chloride. The gas so obtained then passed into a tube containing powdered selenium exposed to sunlight. Under these circumstances the selenium became slowly coated with a deposit of arsenic selenide, arsenic being easily detected in the contents of the tube.

A reaction exactly similar to that occurring when arsine is passed over sulphur exposed to light therefore takes place, although more slowly :—$2AsH_3 + 6Se = As_2Se_3 + 3H_2Se$. Just as in the sulphur experiments, a secondary reaction was found to take place between the liberated hydrogen sulphide and the arsine, resulting in the formation of arsenic sulphide (which deposited on the glass tube) and free hydrogen, so in the selenium experiment, after prolonged exposure to sunlight, the glass tube was found slightly coated with arsenic selenide, due to the reaction of the liberated hydrogen selenide on the arsine :— $2AsH_3 + 3H_2Se = As_2Se_3 + 12H$.

(*b*) *At a Temperature of* 100° C.—Arsine was passed over selenium contained in a U-tube which was immersed in boiling water, and the whole apparatus was screened from daylight. There was no difference in the appearance of the tube at the conclusion of the experiment, and on testing the contents the absence of arsenic was proved, so that arsine is not decomposed by selenium at the temperature of boiling water.

(*c*) *In the Dark.*—Arsine was passed over selenium contained in a tube screened from light. The gas was passed for several hours, but no reaction occurred.

Action of Tellurium on Arsine.

(*a*) *In Sunlight*—Arsine was passed for three hours over powdered tellurium contained in a straight glass tube. At the end of the experiment there was no visible change in the appearance of the tellurium, but on removing it from the tube and heating it in a test-tube, a sublimate formed which, under the microscope, was seen to consist of distinct octahedral crystals. These were dissolved in water, hydrochloric acid and sulphuretted hydrogen were added, and a yellow precipitate of arsenic tri-sulphide appeared. So that, in presence of sunlight, tellurium decomposes arsine.

(*b*) *At a Temperature of* 100° C.—Arsine was passed over powdered tellurium contained in a U-tube protected from light and kept at a temperature of 100° C. After more than three hours treatment the action was stopped and the contents of the tube tested for arsenic as in the preceding experiment, but not a trace of arsenic was detected.

(*c*) *In the Dark.*—The experiment was repeated at the ordinary temperature for the same length of time, but no arsenic could be detected in the contents of the tube.

Action of Selenium on Stibine.

(*a*) *In Sunlight.*—Stibine, prepared in a similar manner to arsine as above, was passed over powdered selenium contained in a glass tube exposed to sunlight. The escaping gas was passed over paper moistened with solution of lead acetate. This very soon blackened, and the stains produced were not rapidly removed either by hydrogen dioxide or hydrochloric acid, and after a time they became orange coloured owing to separation of selenium. During the experiment it was noticed that a brown film slowly formed on the glass tube containing the

* A Paper read before the Manchester Literary and Philosophical Society, March 10, 1908. From the *Memoirs and Proceedings of the Manchester Literary and Philosophical Society*, lii., No. 12.

selenium, showing that in this case also a secondary reaction occurred between the liberated hydrogen selenide and the stibine :—$2SbH_3 + 3H_2Se = Sb_2Se_3 + 12H$.

(*b*) *At a Temperature of* 100° C.—Stibine was then passed over powdered selenium contained in a U-tube immersed in boiling water and screened from light.

As the selenium melts so readily that it might stop the passage of the gas, small portions were placed in the U-tube, separated by plugs of cotton-wool. At the exit a tube was placed containing paper moistened with solution of lead acetate. Soon after the experiment began the lead paper became stained, and eventually was blackened all over. The stains were not removed by treatment with either hydrogen dioxide or hydrochloric acid, so that selenium decomposes stibine at the temperature of boiling water.

(*c*) *In the Dark.*—Stibine was passed over powdered tellurium contained in a straight glass tube and screened from light. A paper moistened with lead acetate solution was placed between the stibine apparatus and the selenium tube, and another at the exit of the apparatus. When the gas had passed for half-an-hour the first paper was quite unaltered, but the second was stained half its length. The experiment was continued and ultimately the second paper was blackened all over. The stains were not removed by hydrochloric acid, so that, even in the dark, stibine is decomposed by selenium. This reaction was so remarkable and unexpected that the experiment was several times repeated with the same result. Of all the hydrides examined in contact with sulphur, selenium, and tellurium, stibine is the only one to be decomposed in the dark at the ordinary temperature, and then only when in contact with selenium.

Action of Tellurium on Stibine.

(*a*) *In Sunlight.*—Stibine was passed over powdered tellurium contained in a straight glass tube and exposed to sunlight. The exit tube contained a paper moistened with solution of lead acetate. This paper became darkened after the prolonged action of the gas, and the stains were not removed when placed in hydrochloric acid, so that stibine is slowly decomposed in presence of tellurium exposed to sunlight.

(*b*) *At a Temperature of* 100° C.—When stibine was passed over tellurium kept at a temperature of 100° C., the lead paper was distinctly stained after the prolonged passage of the gas, and the stains were not removed by hydrochloric acid.

(*c*) *In the Dark.*—In a similar manner, stibine was passed over tellurium, but in the dark and at the ordinary temperature no reaction took place.

The results of the investigation, including those of the previous paper in which sulphur was used, may be tabulated as follows (see next column).

In the previous paper I pointed out that the facility of the decomposition of the hydrides of phosphorus, arsenic, and antimony by means of sulphur when exposed to light, increased with the rise in the molecular weight of these gases, phosphine being very slowly decomposed, arsine more rapidly, and stibine very rapidly. Similarly, it is now to be noted that the action of these hydrides on sulphur, selenium, and tellurium corresponds with the rise in the atomic weights of these elements. This is shown by the comparative rapidity with which the action takes place in each case, stibine being readily decomposed by selenium and more slowly by tellurium exposed to sunlight, whereas arsine reacts on selenium slowly and on tellurium only very slowly. The fact that stibine is decomposed by selenium, even in the dark, is of course a marked exception. I have previously shown that paper coated with sulphur when in contact with stibine and exposed to light, may be used like sensitive paper in photographic printing, the exposed portions becoming orange coloured owing to the deposition of antimony trisulphide, the protected parts retaining the yellow colour of the sulphur. In a similar way paper coated with selenium and kept in contact with stibine may be used as printing paper, but there is, of course, not so great a contrast between the colour of the antimony selenide deposited and the selenium, as there is between the orange-coloured antimony trisulphide and the sulphur.

Element.	Hydride.	Result.
In Sunlight.		
Sulphur	AsH_3	Reacts slowly.
Selenium	AsH_3	Reacts.
Tellurium	AsH_3	Reacts.
Sulphur	SbH_3	Reacts easily.
Selenium	SbH_3	Reacts easily.
Tellurium	SbH_3	Reacts.
At 100° C.		
Sulphur	AsH_3	Reacts slowly.
Selenium	AsH_3	No action.
Tellurium	AsH_3	No action.
Sulphur	SbH_3	Reacts slowly.
Selenium	SbH_3	Reacts slowly.
Tellurium	SbH_3	Reacts very slowly.
In the Dark.		
Sulphur	AsH_3	No action.
Selenium	AsH_3	No action.
Tellurium	AsH_3	No action.
Sulphur	SbH_3	No action.
Selenium	SbH_3	Reacts.
Tellurium	SbH_3	No action.

Action of Arsine on Solutions of Lead.

I have already referred to the action of arsine on solution of lead acetate as preventing the adoption of the lead paper test with this gas. It is surprising that this reaction seems hitherto to have escaped observation, seeing how thoroughly the behaviour of both arsine and stibine with reagents has been investigated. In Gmelin's "Handbook," vol. iv., pages 268 and 335, it is stated on the authority of Simon that arsine and stibine do not act on lead acetate, and I have not found any other statement on the subject. In the course of the experiments above described I found in one case such a rapid blackening of the lead paper, that it occurred to me that it might be due to the arsine itself and not to any reaction on the selenium that was being exposed to it. This led me to remove the selenium tube and allow the gas to act on a fresh paper moistened with solution of lead acetate. This was rapidly darkened and the stain had very much the appearance of papers stained with lead selenide and telluride. These stains are distinguished from those due to lead selenide and telluride by not altering when treated with either strong or dilute hydrochloric acid.

On allowing arsine to bubble through solution of lead acetate contained in a wash-bottle, the liquid soon darkens in colour and a black precipitate separates. Subsequently a black shining mirror forms on the sides of the bottle and adheres firmly to the glass for some days, when it drops off.

I can find nothing in the precipitate but lead and arsenic, but on making quantitative determinations I found such varying proportions of the two elements in different preparations, that I could only conclude that the compound is probably an arsenide of lead but not of definite composition.

Similar experiments with stibine showed that this gas has no action on solution of lead acetate.

NEW APPLICATIONS OF ELECTRO-METALLURGICAL ALLOYS.*

By AD. JOUVE, Ingenieur-Conseil, Ancien preparateur de Chimie à l'Ecole Polytechnique, Directeur de la *Revue d'Electrochimie et d'Electrometallurgie.*

Among the alloys prepared by means of the electric furnace the most important are those containing iron, and particularly the ferro-silicons.

Substances prepared in this way have a percentage of silicon varying from 20 to 75 per cent, the most usual being 20 to 25, 50 to 55, and 70 to 75 per cent.

Their uses are well known to all metallurgists, while the analytical methods for determining them are very numerous, accurate, and wholly without complication, being all based on the solutions of the substance in alkalis in the presence of an oxidising agent such as peroxide of sodium, sodium oxide, or free oxygen.

It is a fact well known to analysts that no methods of analysis are based on the effect of acid, except in the case of hydrofluoric acid, and for this reason ferro-silicons with percentages above 20 per cent are insoluble in acids.

It is this peculiar property, due to silicon alone, which has been employed in an altogether unusual way; *i.e.*, instead of placing ferro-silicons in acids to dissolve them, acids during their preparation may be contained in ferro-silicon apparatus.

Thus, by using high percentage ferro-silicons, apparatus practically unaffected by acids is obtained.

Ferro-silicons are not, however, the only substances which possess this property. For, as this unalterability is due to the silicon, any alloy of a metal with this metalloid will behave in the same way to a greater or less degree according to the nature of the metal. It is evident, for instance, that calcium silicide will be unaffected by acid; the same phenomenon, in fact, occurs, for it is evidently much less affected than metallic calcium would be.

In answer to the question whether the problem of constructing apparatus for the preservation of acids is solved by making use of metallic silicides, it may be said that (1) these substances are very fragile; (2) they contract considerably on cooling, often breaking in the mould when scarcely solid and still at a bright red heat; (3) their melting-point is low compared with the temperatures occurring in ordinary foundry practice—*e.g.*, the second melting in a coke or furnace gas-heated cupola; and (4) certain of these compounds are powerful reducing agents, so that an effect similar to that produced by aluminium may occur. In consequence, fusion in the presence of the air necessary for combustion of the coke or furnace-gas is impossible without considerably lowering the percentage of silicon (especially in rich ferro-silicons—*i.e.*, about 35 per cent) to such a point that sometimes it will become as low as 15 per cent, and thus give alloys which are attacked by moderately strong acids.

Manufacture of Apparatus.—It is not proposed to enter here into complete details of the foundry methods employed, which belong rather to technical metallurgy, and it will simply be shown that by quite special and perfect arrangements, as well as by the correction of the mechanical properties with alloys of different natures, it has been possible to obtain, after six years' research, apparatus of all kinds and descriptions.

The first part of the process consists in obtaining melted metal suitable for pouring into moulds.

The simplest and most used of these methods consists in pouring alloys, melted in that apparatus, directly from the electric furnace into a mould lined with some refractory material. The mixture is then made. In a second method, the alloys obtained from the electric furnace are re-melted in an oil, or in certain cases in a crucible furnace and then cast into ingots.

Once the liquid metal is obtained, metals for corrective purposes are added. The number and nature of these is practically infinite for any particular case. They are added directly to the metal in the casting-ladle, usually with aluminium, which raises the temperature of the bath and increases the fluidity.

When the ladle is ready it is used mechanically, by a travelling crane, say, and carried quickly, to prevent cooling, to the mould. The pouring is effected by ordinary means without any special precautions.

The casting is removed as soon as it solidifies, is turned out into the warm casting sand, and thus annealed at a bright red heat. It is then ready for use.

Resistance to Acids.—The resistance of the metallic alloys of silicon, which have been called "Métillures," to acids is quite interesting, as the following examples will show. Nitric acid, even as a vapour, such as is obtained at the exit of a bisulphate retort or when mixed with nitrous acid, does not affect them at all. A striking example of this is given by a pipe which has been submitted for nearly five years to the daily passage of 660 lbs. of nitric acid vapour at temperatures varying from 150° to 200° without its loss in weight exceeding a few decigrms. in a total weight of a score of kilogrms. This loss occurred quite at the beginning of the period, and was probably due to a few impurities remaining on the inner surface of the pipe after fusion.

Sulphuric acid has still less effect, and acids of strength from 20 to 25, known as *petites eaux*, may be concentrated in vessels of this kind by direct heating and continuous evaporation.

Hydrochloric acid is analogous in its action, and use has been made of pipes of the above material for carrying and condensing its gases.

Acetic acid and the mixture produced by treating calcium acetate with sulphuric acid are without effect on "Métillures," even in the presence of air.

Resistance to other Reagents.—An important English firm has obtained the following figures relating to the resistance of "Métillures" to the action of cyanides, sulphocyanides, and prussic acid. The results are very remarkable, for they show both that the unattackability increases with the percentage of silicon, while for a suitable percentage it is practically negligible.

"Métillures" (20 per cent Nitric Acid).

No.	Weight in grms.	Change in weight. After 24 hours.	Reaction with sulphocyanide.
1.	6·4410	−0·0392	Violent.
2.	5·1669	−0·0047	Slight.
3.	8·1524	−0·0037	Moderate.
4.	14·5013	+0·0012	Moderate.
5.	1·7060	−0·0716	Very violent.
6.	1·4941	−0·0034	Very slight.
		After 48 hours.	
1.	6·4018	−0·0207	Violent.
2.	5·1622	−0·0006	Very slight.
3.	8·1487	−0·0032	Moderate.
4.	14·5025	+0·0045	Slight.
5.	1·6344	−0·0323	Violent.
6.	1·4907	−0·0007	None.

Métillures (13 per cent Sulphocyanide of Aniline).

No.	Weight in grms.	Change in weight. After 24 hours.	Reaction with potassium ferricyanide.
1.	6·3811	−0·0387	Very violent.
2.	5·1616	−0·0065	Moderate.
3.	8·1455	−0·0455	Violent.
4.	14·5070	+0·0055	Slight.
5.	1·6021	−0·0186	Violent.
6.	1·4900	−0·0160	Very slight.
		After 48 hours.	
1.	6·3430	−0·0675	Very violent.
2.	5·1551	−0·0219	Moderate.
3.	8·1000	−0·0433	Violent.
4.	14·5125	−0·0063	Slight.
5.	1·5835	−0·0216	Violent.
6.	1·4740	−0·0011	Very slight.

* A Paper read before the Faraday Society, June 23rd, 1908.

Under the same conditions, iron or any other metal in commercial use would be rapidly attacked, thus preventing its employment. The apparatus constructed up to the present time is extremely varied both in form and dimensions, ranging from evaporating dishes, troughs, right-angled bends, elbows, worms, ventilators, &c., and in the other from those weighing about 20 lbs. and having a diameter of 1 ft. to those weighing over one ton and measuring 7 ft. across, as well as pipes with a diameter of 8 ft. and weighing about three-quarters of a ton.

To sum up, it may be said that the production of a material capable of resisting acids is an accomplished industrial fact, and the principal desiderata are realised. It remains, however, to remedy the unmalleability of these substances. This question is actually being studied, and a solution seems probable in the near future.

As electro-metallurgists we may congratulate ourselves that without the electric furnace, which alone permits sufficiently high temperatures for the reduction of silicon or silicates by carbon to be obtained, the high percentage alloys of metals with silicon could not have been manufactured.

Further, without the electric furnace the fusion of high-percentage silicides would not have been possible. For when the percentage exceeds 50 the temperature of a coke or gas furnace is not sufficient to permit these alloys to be cast, not because they will not run, but because their specific heat is too low for the metal to be poured. In fact, it will not leave the crucible, but forms a pasty mass.

This latest conquest of the electric furnace has come at a most suitable time. For platinum, the most stable industrial metal, is becoming more and more rare, while its price is increasing by leaps and bounds, making its employment a matter of greater and greater difficulty.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 7).

General Discussion (continued).

6. THE presence of beryllium does not involve any complication, since it goes with the aluminium in the process just referred to, and can be separated from it as described below. When, however, uranium and vanadium are to be provided for, this process is entirely inadequate, for upon the addition of ammonium hydroxide after acidifying, vanadium divides between the filtrate and precipitate wherever uranium is present, owing to the insolubility of uranyl vanadate; uranium itself will divide owing to the presence of H_2O_2 formed on acidifying the sodium peruranate solution; and vanadium, when present in large amount, again divides upon the addition of barium chloride to the acetic acid solution, owing to the slight solubility of barium vanadate; finally, vanadium interferes with the test for zinc with H_2S in acetic acid solution, since a precipitate of sulphur is always formed, and sometimes one of black vanadium sulphide. Moreover, the uranium precipitate obtained with ammonium hydroxide will, in general, be mixed with aluminium, beryllium, and vanadium, so that the difficult part of the separation still remains to be accomplished. After much experimenting, guided by the conception that under proper conditions of alkalinity it might be possible to separate the more basic elements, zinc, aluminium, and beryllium, from those present as constituents of acid radicals, chromium, uranium, and vanadium, it was finally found that this could be accomplished in a hot solution of sodium hydrogen carbonate, provided care be taken to prevent loss of carbon dioxide by heating the solution in an open vessel only to 90°, or better in a closed bottle to 100°. Under these conditions the separation is a fairly satisfactory one. A small amount of uranium may, however, be carried down almost completely when a large amount of aluminium or beryllium is present, making it necessary to test for uranium in the precipitate. Moreover, when uranium and vanadium are simultaneously present, each in large quantity (about 100 mgrms.) some uranyl vanadate precipitates, but a large quantity of both elements remains in the solution, so that their detection is not interfered with.

7. The separation of the zinc from the aluminium and beryllium in the precipitate is readily effected by dissolving it in hydrochloric acid, and adding a small excess of ammonium hydroxide. For the separation of the aluminium and beryllium from each other we studied what seemed to be the two most promising methods mentioned in the literature. The first of these was that described by Parsons and Barnes (*Journ. Am. Chem. Soc.*, 1906, xxviii., 1589), which consists in boiling for a short time a solution of the two elements to which enough sodium hydrogen carbonate is added to make a 10 per cent solution, whereby aluminium is precipitated and beryllium dissolved. We found, however, that though this method is satisfactory for the detection of beryllium when a moderate amount of aluminium is present, yet with a large quantity of aluminium (say, 100 to 500 mgrms.) 2 to 5 mgrms. of beryllium are almost completely retained in the precipitate, which may cause it to escape detection, and which at any rate leads to an incorrect estimate of its quantity. The second method investigated was that proposed by Havens (*Zeit. Anorg. Chem.*, 1898, xvi., 15), which consists in saturating a solution of the chlorides of the two elements in a mixture of ether and strong hydrochloric acid with hydrogen chloride gas, whereby aluminium is precipitated and beryllium remains in solution. It was found that this method gives entirely satisfactory results; even 0·5 mgrm. of aluminium is precipitated as $AlCl_3.6H_2O$, provided care be taken to use a sufficient proportion of ether and to saturate completely with the gas; even 100 mgrms. of beryllium remain wholly in solution, and 0·5 mgrm. is not carried out with a large quantity of aluminium. This method was therefore adopted for the separation, it being supplemented by a confirmatory test for beryllium based on the process of Parsons and Barnes.

8. The separation of the chromium, vanadium, and uranium, which are present together in the filtrate from the sodium hydrogen carbonate precipitate, also required much investigation. It was soon decided that there was more promise of effecting a separation of the first two of these elements in the state of chromate and vanadate, in which they already exist, than in a lower stage of oxidation; and it was found that the lead salts differed sufficiently in solubility in nitric acid to enable 0·5 mgrm. of chromium to precipitate while retaining 100 mgrms. of vanadium in solution. A separation based on this fact was therefore adopted. The excess of lead added is subsequently removed by saturating the filtrate with hydrogen sulphide. To avoid the addition and removal of lead when chromium is absent, a preliminary test for chromate with hydrogen peroxide is introduced. The uranium is separated from the vanadium (after oxidation to the vanadic state) by precipitating it as uranyl ammonium phosphate in acetic acid solution—a method that was found to give satisfactory results for the limiting case of a large proportion of uranium and a small proportion of vanadium, and also in the converse case. Vanadium is tested for by making the filtrate strongly alkaline with ammonia and saturating with hydrogen sulphide, whereby a violet-red solution of a vanadium sulpho-vanadate is formed.

9. We will next consider the analysis of the precipitate produced by sodium peroxide, which contains the manganese, iron, nickel, cobalt, thallium, titanium, and zirconium, the alkaline earth elements, and possibly phosphate. The main problems connected with this were the

* From the *Journal of the American Chemical Society*, XXX., No. 4.

separation of the manganese from the other elements, that of the alkaline earth elements from phosphate, and that of the titanium and zirconium from the iron and from each other.

10. In almost all schemes of qualitative analysis it is thought sufficient to test a portion of the precipitate for manganese by fusing it with sodium carbonate or by boiling it with lead dioxide and nitric acid, without isolating the manganese. These colour tests give, however, but little idea of the quantity of the element present. Moreover, aside from this objection, the large number of the elements contained in the precipitate in this scheme makes their separation necessary. The one reaction of manganese which seemed in every way suited for this purpose is that frequently employed in iron and steel analysis, consisting in the conversion of the manganese into the dioxide by the action of chloric acid and concentrated nitric acid. For this is not only a behaviour highly characteristic of this element, but, since the separation is carried out in a strongly acid solution, it might be anticipitated that the other elements, which are not oxidisable to insoluble peroxides, would not be retained in the precipitate to an important extent. Our experiments have shown that this is, in general, the case, but one exception has been discovered. It has been found, namely, that titanium, which is quadrivalent like manganese in the dioxide, when present even in considerable quantity (up to 50 mgrms.) may be completely precipitated with a large quantity of manganese (500 mgrms.), and that a large proportion, though not all of the zirconium is likewise carried down. The method is, however, otherwise so satisfactory that we have adopted it, special provision being made for this unusual case in a way that need not be described here. The procedure consists in dissolving the whole sodium peroxide precipitate in strong hydrochloric acid, in evaporating with excess of nitric acid, adding concentrated nitric acid and potassium chlorate, heating, and filtering off the manganese dioxide on an asbestos filter.

11. Owing to the fact mentioned above, that zinc is carried down in considerable quantity by manganese in the Na_2O_2 precipitation, experiments were made with the view of previously removing manganese by introducing this chloric acid procedure at the beginning of the analysis of this group. It was found, however, that vanadium, which is not carried down in the sodium peroxide procedure, is, like titanium, precipitated in large quantity with the manganese in the chloric acid procedure. It was found also that some zinc (1 to 4 mgrms.) may be carried down completely in the Na_2O_2 precipitate by iron, nickel, and cobalt, so that it would be still necessary to provide for the detection of zinc in the iron group. For these reasons, it is evidently best to have the sodium peroxide precipitation precede the treatment with chloric acid.

(To be continued).

Cæsium Dicalcium Sulphate.—J. D'Ans.—To prepare cæsium dicalcium sulphate a solution of gypsum is added to a solution of cæsium sulphate. The salt formed resembles the ammonium and rubidium salts when examined under the microscope. It does not crystallise with water and is very stable, and the stability increases as the temperature rises. The compounds of alkali sulphates with calcium sulphate which have been prepared have the following formulæ:—

$K_2Ca(SO_4)_2.H_2O$	$Rb_2Ca(SO_4)_2.H_2O$
	$Rb_2Ca_2(SO_4)_3$
$K_2Ca_5(SO_4)_6.H_2O$	
$(NH_4)_2Ca(SO_4)_2.H_2O$	
$(NH_4)_2Ca_2(SO_4)_3$	$Cs_2Ca_2(SO_4)_3$
$(NH_4)_2Ca_5(SO_4)_6.H_2O$	

As the atomic weight of the alkali sulphate increases the limits of the temperature of stability of the double sulphates become lower. Thus potassium syngenite is stable above 100°, rubidium syngenite only up to about 40°, ammonium dicalcium sulphate above 80°, and cæsium dicalcium sulphate below 0°.—*Berichte*, xli., No. 8.

THE CHEMICAL SOCIETY.

PROPOSAL TO ADMIT WOMEN TO THE FELLOWSHIP.

THE following correspondence has been forwarded to us for publication:—

1. *Copy of a Letter from the Secretaries to the Fellows of the Chemical Society.*

Burlington House, Piccadilly, W., June, 1908.

DEAR SIR,—The Council of the Chemical Society have received a petition praying for an inquiry as to the views of the Society as a whole on the question of admitting women to the Fellowship.

Accordingly, the Council invite very careful consideration of the following brief statement of the chief arguments which have been used both for and against the admission of women.

It is proper to point out that the Council were advised on a former occasion by Counsel that there is some doubt whether, under the existing Charter, women are admissible as Fellows; it is also well to remember that if admitted to this status, women would be eligible, like other Fellows, for a seat on the Council and to hold office.

Those who support the view that steps ought to be taken in order to provide for the admission of women to the Fellowship point out—

1. That the petition has been signed by 312 Fellows (including 10 Past Presidents, 12 Vice-Presidents, and 29 Members of Council, past and present), among whom are 33 Fellows of the Royal Society and the Professors of Chemistry or Heads of Chemical Departments of nearly all the most important Universities and Colleges in the country.

2. That a number of women are now devoting themselves to the science of chemistry, the study of which it is the chief object of the Chemical Society to promote. It is shown that they are capable of independent work by the facts stated in the petition: moreover, in the *Transactions* from January to May this year there are four papers by women authors independently of others in which they appear as joint authors.

3. The Chemical Societies of Berlin and America and the Institute of Chemistry admit women to full privileges of membership. The Chemical Society itself has placed the name of Madame Curie among those of its Honorary Members.

4. A small number of women chemists attend the meetings of the Society regularly as visitors, and no inconvenience has arisen from their presence. They ask that they should be admitted in order that, in addition to exercising the voting powers, they may use the Library and receive the publications of the Society on the same terms as Fellows; at present they have to pay the higher price of publication charged to the general public.

5. The Society numbers over 2800 Fellows, while the number of women desiring admission at the present time is about 20; this number will probably increase as time goes on, but judging by the experience of other Societies, it is not likely greatly to exceed that number in the present generation. Should the number rise to as many as 50, their numerical strength would remain wholly insignificant in regard to the conduct of the Society's affairs; consequently, any fear that female influence might hereafter dominate the Council, or even that one woman might be elected to the Council except only in recognition of her scientific ability, is not worth consideration. As to the undesirability of substituting one sex for another in offices of employment, little argument can be based on such a consideration so long as men continue to hold many of the teaching posts in women's colleges.

6. There is reason to believe that in the event of a decision in favour of applying for a supplemental Charter, the cost, or a large part of it, would be borne by the women chemists and their friends.

On the other hand, those who are unwilling to admit women to the Fellowship of the Chemical Society urge that—

1. The expense, probably amounting to several hundred pounds, which would be incurred by the Society if a supplemental Charter is necessary, is not justified by the small number of new Fellows likely to seek admission.

2. It may be gravely doubted whether the deliberate encouragement of women to enter the chemical profession would not operate unfavourably on women themselves in view of the arduous nature of chemical work.

3. Although it is true, as urged by supporters of the petition, that the number of women seeking admission is small, it is also claimed as a reason for admission that the volume of chemical work contributed by women is increasing rapidly. So long as the women who desire admission remain in such disproportionate minority, there will be an inevitable—although, of course, involuntary—tendency to overestimate their contribution to chemical knowledge and practice. An illustration of this principle may fairly be drawn from the petition itself, where figures are adduced to show that during the past thirty-five years women have been associated with 103 papers contributed to the *Transactions* of the Society, but it is not stated, though equally true, that while the total number of papers printed during that period exceeds 3400 only twenty-three are in the names of women alone.

4. Even assuming that these twenty-three contributions were independent of masculine inspiration, it may be questioned whether women have, as a group, shown marked aptitude for chemical pursuits, particularly when it is recalled that the authors in question have worked almost exclusively in collegiate institutions.

5. Moreover, by being welcomed as guests to the Society, women have been able to enjoy that chemical atmosphere and intercourse which Fellowship of the Society involves.

6. As regards the admission of Madame Curie to Honorary Membership, it must be borne in mind that Honorary and Foreign Members have no voting powers, and are not eligible for office.

7. Briefly stated, the position of those unfavourable to the admission of women is that, whilst gladly offering to those women who already have become chemists measures which would give them the benefits derived from attendance at the meetings, they deem it inexpedient publicly to encourage women to adopt chemistry as a professional pursuit, since such a course would tempt them into a career in which they may ultimately not find employment in view of the already over-crowded state of the profession.

Other minor considerations might be mentioned on both sides, and rejoinders to all the above statements are generally obvious. The Council therefore hope that every Fellow will carefully consider the arguments on both sides, and give them the weight which each, respectively, appears in his judgment to deserve.

You are requested, after making the necessary deletion, to return the enclosed ballot paper to the Secretaries in the accompanying envelope, which envelope must be endorsed with your signature. *Unsigned envelopes and signed ballot papers will be invalid.*

The ballot will close on the first day of October next. —We are, Sir, yours faithfully,

M. O. FORSTER,
ARTHUR W. CROSSLEY, } Hon. Secretaries.

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2. *Copy of a Letter from Past Presidents of the Chemical Society accompanying the communication of the Petition to the Fellows.*

Oxford, 1st July, 1908.

DEAR SIR,—In the letter, which has been sent by the Secretaries of the Chemical Society to every Fellow, on the question of admitting women to the Fellowship, reference is made to a petition received by the Council. We think it desirable that the petition itself, which is not only a petition but a declaration of opinion on the part of a large body of the Fellows of the Society, should be made known generally, together with the names of those by whom it was signed. Accordingly we enclose herewith a copy of the petition as presented to Members of the Council.

Before the issue of the voting papers the attention of the Secretaries was called to an ambiguity which is due to the introduction of the words "the full rights and privileges of." We suggest that those Fellows who are in favour of the admittance of women to the Fellowship, and not only to "the full rights and privileges of the Fellowship," should strike out these words. It would clearly be possible, however unreasonable, to create a class who, though not actually Fellows, were admitted to all the rights and privileges of the Fellowship. That such a class should be created is not the question which the Council were asked and consented to put.—We are, Sir, yours faithfully,

(Signed)

WILLIAM ODLING, T. E. THORPE,
WILLIAM CROOKES, J. EMERSON REYNOLDS,
A. VERNON HARCOURT, WILLIAM A. TILDEN,
HENRY E. ROSCOE, R. MELDOLA,
HUGO MÜLLER, ALEX. CRUM BROWN.
W. J. RUSSELL,

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3. *Copy of the Petition.*

TO THE PRESIDENT AND COUNCIL OF THE CHEMICAL SOCIETY.

GENTLEMEN,—We, the undersigned Fellows of the Chemical Society, being of opinion that the time has come when the Fellowship of the Society should be rendered accessible to women, request the Council to take such steps as may appear desirable to ascertain the wishes of the Society as a whole in regard to this question.

We understand that there is now an appreciable and increasing number of women of University training engaged in advanced teaching, and in original investigation in chemistry, who desire admission to the privileges of the Fellowship, and as the Chemical Society was founded for the advancement of Science, it seems to us neither just nor expedient that a body of highly qualified workers should be excluded solely by reason of sex.

From the following table, compiled from the Society's journal of the past thirty-five years, it will be seen that the number of papers contributed either alone, or jointly, by women is increasing rapidly:—

	Number of Papers published in—	
	Proceedings.	Transactions.
1873—82..	2	2
1883—92..	7	7
1893—02..	45	33
1903—07..	66	61

We may further point out that not only have women contributed original memoirs to these publications, but they have rendered valuable service to the Society as abstractors and in the compilation of the Indexes.

As is well known, the Chemical Societies of Berlin and America, the Society of Chemical Industry, and the Faraday Society admit women on the same terms as men, and our Society has found a place for Madame Curie among the Honorary and Foreign Members; we consider, therefore, that the restriction should be removed under which the Chemical Society denies to women chemists the advantages extended to them by the sister Societies at home and abroad.—We are, Gentlemen, your obedient servants,

(Signed)

Robt. T. Abell, R. H. Adie, John Allan, Albert E. Andrews, A. Angel, Paul S. Arup, P. C. Austin, G. H. Bailey, H. B. Baker, Horatio Ballantyne, Edward C. Cyril Baly, George Barger, Wm. Barlow,

M. Barrowcliff, P. Phillips Bedson, W. H. Bentley, Leslie H. Berry, Edward Bevan, Geo. S. Blake, Bertram Blount, B. H. Bowles, H. L. Bowman, Joseph Brierley, James S. Broome, Alex. Crum Brown, David Brown, William Burton, J. C. Cain, Kenneth Caldwell, Wm. Caldwell, Hugh C. H. Candy, H. C. H. Carpenter, John A. Carpenter, Francis H. Carr, John Castell Evans, F. W. Caton, R. M. Caven, D. L. Chapman, A. H. Church, H. T. Clarke, G. W. Clough, J. Bernard Coleman, A. J. Cook, A. H. Coote, H. F. Coward, J. C. Crocker, Holland Crompton, William Crookes, C. F. Cross, J. K. Crow, Alfred V. Cunnington, W. A. Davis, H. M. Dawson, George Dean, William S. Denham, Cecil H. Desch, Edward Divers, George Dixon, Harold B. Dixon, Leonard Dobbin, F. W. Dootson, Thos. R. Duggan, Bernard Dyer, Lewis Eynon, T. W. Fagan, Thomas Fairley, E. H. Farr, Chas. A. Fawsitt, Charles E. Fawsitt, Ernest Feilmann, H. J. H. Fenton, Alex. Findlay, Horace Finnemore, W. W. Fisher, John Foggie, John S. Ford, Gilbert J. Fowler, J. J. Fox, Francis Francis, H. Frankland, Percy F. Frankland, Angus Fraser, Robt. C. Gale, Edward Gardner, J. A. Gardner, G. H. Gemmell, C. S. Gibson, John Gibson, J. F. H. Gilbard, J. N. Goldsmith, F. Gossling, Jas. Grant, R. W. Gray, Thomas Gray, A. G. Green, A. J. Greenaway, Robt. G. Grimwood, P. Haas, A. D. Hall, W. A. Handcock, F. W. Harbord, A. Vernon Harcourt, Arthur Harden, H. Harding, E. F. Harrison, Fred T. Harry, W. Beaumont Hart, E. G. J. Hartley, H. B. Hartley, W. N. Hartley, Jack V. J. Hayman, Otto Hehner, G. G. Henderson, James Hendrick, J. T. Hewitt, T. P. Hilditch, W. R. Hodgkinson, George T. Holloway, Alfred Holt, jun., E. Grant Hooper, F. G. Hopkins, Edward Horton, Alfred G. Howard, Jul. Hübner, A. K. Huntington, G. Nevill Huntly, W. H. Hurtley, A. Hutchinson, Ernest W. Jackson, Herbert Jackson, T. Campbell James, F. R. Japp, E. Jones, Francis Jones, H. O. Jones, A. M. Kellas, M. Kemp-Welch, Frank E. King, Frank W. G. King, J. Falconer King, F. Stanley Kipping, Patrick H. Kirkaldy, Harley F. Knight, W. R. Simpson Ladell, F. E. E. Lamplough, G. Druce Lander, Arthur Lapworth, A. T. Larter, R. T. Lattey, Alexander Lauder, H. D. Law, J. B. Leathes, Frederic H. Lees, R. Lessing, Edward W. Lewis, W. J. Lewis, J. Lewkowitsch, Arthur R. Ling, G. D. Liveing, J. P. Longstaff, T. M. Lowry, W. Lowson, G. Roche Lynch, John S. MacArthur, Hamilton McCombie, Alex. McKenzie, John E. Mackenzie, Herbert McLeod, Alan Macmullen, W. Maitland, Arthur Marshall, Hugh Marshall, N. H. Martin, F. E. Matthews, Raphael Meldola, A. M. Meldrum, A. E. Middleton, Henry A. Miers, J. Bruce Miller, Edmund J. Mills, W. H. Mills, Chas. W. Moore, T. S. Moore, Hy. Forster Morley, Richard J. Moss, A. de Mouilpied, Hugo Müller, M. M. Pattison Muir, Thos. J. Murray, D. H. Nagel, S. S. Napper, F. H. Neville, B. E. R. Newlands, W. E. Oakden, A. O'Farrelly, Kennedy J. P. Orton, John Parkin, S. Parrish, T. S. Patterson, S. J. Peachey, F. Mollwo Perkin, W. H. Perkin, W. H. Perkins, E. P. Perman, James C. Philip, G. S. Philpotts, Robert H. Pickard, Sam. S. Pickles, D. R. Pinnock, George P. Pollitt, James H. Pollok, W. J. Pope, F. B. Power, Henry R. Proctor, C. Proctor, Thomas Purdie, Frank Lee Pyman, J. E. Purvis, G. G. Quinn, L. G. Radcliffe, A. A. Read, Walter F. Reid, P. Remfrey, J. Emerson Reynolds, Wm. C. Reynolds, Edmund M. Rich, F. G. Richards, H. D. Richmond, J. H. Robbins, James Roberts, junr., Wm. Robertson, Henry H. Robinson, R. Rodger, Harold Rogerson, Henry E. Roscoe, S. Ruhemann, A. R. Runeckles, E. J. Russell, W. J. Russell, J. H. Ryffel, Alfred Gordon Salamon, A. H. Salway, Henry J. S. Sand, H. C. Sayer, S. B. Schryver, W. J. Sell, George Senter, A. Humboldt Sexton, W. B. Shaw, W. F. J. Shepheard, N. V. Sidgwick, J. L. Simonsen, F. S. Sinnalt, Sidney Skinner, Arthur Slator, Samuel Smiles, Clarence Smith, Norman Smith, Arthur Smithells, Frederick Soddy, J. F. Spencer, F. E. Stead, Jas. Steger, T. Stenhouse, Thos. Stevenson, A. W. Stewart, F. Henry Streatfeild, R. de Fleming Struthers, Geo. Stubbs, J. J. Sudborough, R. R. Tatlock, R. L. Taylor, W. H. Taylor, F. Thomas, Claude M. Thompson, John Millar Thomson, R. T. Thomson, Leonard Temple Thorne, J. F. Thorpe, T. E. Thorpe, Thomas Tickle, W. A. Tilden, C. K. Tinkler, F. D. Toyne, Frank Tutin, Thos. Tyrer, V. H. Veley, E. W. Voelcker, John Wade, A. F. Walden, James Walker, George Stanley Walpole, George J. Ward, H. Edmeston Watson, J. Adam Watson, H. E. Watt, John Watts, Emil A. Werner, E. Wheeler, R. Whymper, W. H. Willcox, W. Williamson, N. T. M. Wilsmore, Leonard P. Wilson, T. B. Wood, J. Woodward, H. Wren, W. Palmer Wynne, Jos. Yates, George Young, J. Rymer Young, Sydney Young.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 18th, 1908.

Sir William Ramsay, K.C.B., F.R.S., President, in the Chair.

Messrs. F. W. Caton, H. F. Coward, and E. J. Watkins were formally admitted Fellows of the Society.

The President made a short communication with reference to the Seventh International Congress of Applied Chemistry, to be held in London at Whitsuntide, 1909 (May 27th to June 2nd), under the Hon. Presidency of Sir Henry Roscoe, he himself being the acting President. The King has consented to be Patron, and the Prince of Wales Vice-Patron; it is hoped that His Royal Highness will be able to open the Congress. The meetings will be held in the University Buildings, in the Imperial College of Science, and in the Central Technical College. There are special sections for (1) Analytical Chemistry; (2) Inorganic Chemistry and Allied Industries; (3) Metallurgy and Mining—Explosives; (4) Organic Chemistry and Allied Industries; (5) Industry and Chemistry of Sugar; (6) Starch Industry; (7) Agricultural Chemistry; (8) Hygiene, Medical, and Pharmaceutical Chemistry—Bromatology; (9) Photographic Chemistry; (10) Electrical and Physical Chemistry; and (11) Law, Political Economics, and Legislation with reference to Chemical Industries, of which Presidents, Vice-Presidents, and Secretaries have already been appointed.

A considerable sum has already been collected privately to meet the expense of the Congress, and a general appeal will be issued shortly. The provisional Programme of the week has been drawn up; it comprises the opening meeting in the Albert Hall, a conversazione at the Natural History Museum, a banquet at the Crystal Palace, and lectures by Prof. Haller, Prof. Otto Witt, Prof. Nasini, and Sir Boverton Redwood. Sectional meetings, at which papers will be read, will be held daily during the meeting. Committees have been formed in foreign countries to organise the work of the sections, and an executive Committee has charge of the arrangements at home. It is anticipitated that the Congress will be largely attended; meetings in Paris, Berlin, and Rome were attended by about 3000 chemists. The Hon. Secetary is Mr. William Macnab, of 10, Cromwell Cresent, S.W.

Certificates were read for the first time in favour of Messrs. William Colet Birch, B.A., 22, Glazbury Road, W. Kensington, W.; John Griffin, B.A., 7, Carlton Terrace, Batley; Joseph Albert Hughes, School of Mines, Charters Towers, Queensland; Thomas Ernest Hull, 6, Selborne Terrace, Greenbank Darlington; James Colquhoun Irvine, Ph.D., D.Sc., Dunmurry, Rathelpie, St. Andrews; Frederick Martin Kay Menzies, M.D., F.R.C.P., 79, Davies Street, Berkeley Square, W.; Howard Houlston Morgan, B.Sc., Technical College, Auckland, New Zealand.

A certificate has been authorised by the Council under By-law I. (Par. 3) in favour of H. V. Krishnayya, B.A., Mallesavaram, Bangalore, Mysore, India.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—William Henry Ballantyne, B.Sc.; Edward Morris Bevan; Donald Francis Blyther, B.Sc.; James Brown; Charles Duncan Cooper; John Robert Cowburn; James Hill Fairweather; Kaufman George Falk, B.Sc., Ph.D.; Frank William Crossley-Holland; Norman Holland; Henry Brougham Hutchinson, Ph.D; James Kirkman King, B.A.; Lionel Gordon Larmuth; Henry Robert Lyell; Alfred Ash Meggitt, B.Sc.; Stanley Allen Warrington Okell; Haradhan Ray, M.A.; William Readwin; Clement Thomas Rutter; James Edward Southcombe, M.Sc.; John Day Speed; Alexander Mackintosh Stewart; Edward Stokes, B.A.; Hari Prasad Vidyant, M.A.; Herbert Horace Ward; Charles Harold Wright, B.A.; Harry James Yates; Charles Robert Young, B.Sc.

Of the following papers, those marked * were read:—

"143. "*The Thermal Decomposition of Hydrocarbons. Part I. [Methane, Ethane, Ethylene, and Acetylene].*" By William Arthur Bone and Hubert Frank Coward.

The results of a systematic investigation of the modes of decomposition of the four hydrocarbons at temperatures between 500° and 1200° were described, The theories advanced by Berthelot, V. B. Lewes, and others were critically examined, and doubts raised as to the importance of acetylene as a factor in such processes.

Methane, which is by far the most stable of the four hydrocarbons, and a principal product of the decomposition of the other three, decomposes for the main part *directly* into carbon and hydrogen. The decomposition is, within the range of temperature investigated, a surface effect, and the carbon deposited is of a peculiarly hard and lustrous type. The large quantities of methane formed during the decomposition of the other three hydrocarbons can only be explained on the supposition that "residues," such as :CH and :CH_2 (which may be considered to have a momentary separate existence during the dissolution process), are directly "hydrogenised" in an atmosphere rich in hydrogen. The assumption of the momentary formation of such "residues" (or the tendency to give rise to them, which actually becomes operative at moderately high temperatures) as the result of the initial dissolution of the hydrocarbon molecule, renders it possible to formulate a rational scheme of the chief causes at work, and their modes of operation in each particular case.

It may be supposed that these "residues" are capable of (*a*) forming $H_2C{:}CH_2$ or HC:CH as the result of encounters with other like residues, or (*b*) breaking down directly into carbon and hydrogen, or (*c*) combining with hydrogen to form methane, and that all three possibilities may be realised simultaneously in proportions dependent on the temperature and the amount of hydrogen present. The scheme of decomposition of the three hydrocarbons (omitting secondary "polymeric" changes, which are essentially low temperature effects) is as follows:—

$$\underset{\text{Ethane.}}{\overset{H\,H}{\overset{|\;\;|}{H\cdot\dot{C}\cdot\dot{C}\cdot H}}} = [2(:CH_2) + H_2] = \begin{cases} (a)\ C_2H_4 + H_2. \\ (b)\ 2C + 2H_2 + H_2. \\ (c)\ \textit{plus}\ H_2 = 2CH_4. \end{cases}$$

$$\underset{\text{Ethylene.}}{\overset{H\,H}{\overset{|\;\;|}{H\cdot\dot{C}{:}\dot{C}\cdot H}}} = [2(:CH) + H_2] = \begin{cases} (a)\ C_2H_2 + H_2. \\ (b)\ 2C + H_2 + H_2. \\ (c)\ \textit{plus}\ 2H_2 = 2CH_4. \end{cases}$$

$$\underset{\text{Acetylene}}{H\cdot C{:}CH} = [2(:CH)] = \begin{cases} (b)\ 2C + H_2. \\ (c)\ \textit{plus}\ 3H_2 = 2CH_4. \end{cases}$$

Polymerisation to C_6H_6, &c.

Discussion.

Prof. Armstrong said that the conditions under which acetylene was produced were undoubtedly peculiar; he had sought for it in vain in the oil gas produced by cracking petroleum. If formed in the process, it underwent change very readily. It was by no means necessary to assume that the benzene which was produced was formed from acetylene. He could not agree with Dr. Bone as to the influence of surfaces in the changes studied; he thought they must play a predominant part. He was in agreement with him, however, as to the manner in which methane was formed; that is to say, that it was formed by the hydrogenation of fragments.

Dr. Divers asked whether the production of methane from acetylene at 800°, in presence of large excess of hydrogen, was not sufficiently accounted for as being the result of the union of the acetylene and hydrogen with decomposition of the product into methane, without making the assumption of the temporary existence of the substance CH?

Prof. Mills referred to the great importance of the author's investigation, especially in connection with the manufacture of coal-gas. He thought the experiments showed that hydrogen (probably on account of its less density) protected ready-made methane better than nitrogen did. Hydrocarbons, such as CH and CH_2, if they could possibly be formed, would, in his opinion, be certain to polymerise (like acetylene, for instance), and, in their polymerised state, it would be extremely unlikely that hydrogen would combine with them.

Prof. Bone, in reply, said he was clearly of the opinion that, whilst the decomposition of methane was, within the range of temperature investigated, entirely a surface effect, in the other three cases surface played a much less prominent part. No one, he thought, could witness the sudden decomposition of acetylene at high temperature with its accompaniment of incandescence without admitting that the chemical change took place throughout the whole mass of the gas and was not confined to the layer in contact with the hot walls of the containing vessel. Time did not permit him to cite all the evidence in favour of the hypothesis that such residues as :CH_2 and :CH are actually formed during these decompositions; but he thought that when Dr. Divers and Prof. Mills had had the opportunity of studying the whole of the facts brought to light during the investigation, their doubts on that point would largely disappear. He himself felt convinced that, in the cases of ethane and ethylene, the collapse of the molecule was preceded by the elimination of hydrogen in the manner stated in the paper.

*144. "*The Rusting of Iron.*" By William Augustus Tilden.

An account was given of a series of experiments from which the following conclusions are drawn.

1. Oxygen or air together with liquid water are alone necessary to produce rusting without the assistance of carbonic acid, failure on the part of other experimenters to produce rusting in iron prepared by treatment with chromic acid being probably due to "passivity."

2. Water alone in the absence of oxygen attacks iron slowly, producing a film of what is probably ferrous hydroxide, as it is wholly converted into brown rust on admission of pure oxygen. In this connection, the experiments of Whitney (*Journ. Am. Chem. Soc.*, 1903, xxv., 298), repeated and approved by Walker, Cederholm, and Bent (*Journ. Am. Chem. Soc.*, 1907, xxix., 1255), were confirmed.

3. Iron rust even when very old and constantly exposed to the weather always contains ferrous oxide.

4. The process of rusting is due, in the first instance, to electrolytic action promoted in all ordinary cases by the existence of carbonic acid in water exposed to the air, and by the presence in iron of various compounds of carbon, silicon, phosphorus, and sulphur. That electrolytic action does occur locally in all commercial iron, whether malleable iron, grey or white cast-iron, or steel immersed in water, is shown by the use of the "ferroxyl" indicator (phenolphthalein and ferricyanide) introduced by Walker and his colleagues, and in other ways. In ordinary rusting, the process is also assisted by the depolarising action of the layer of ferric oxide or hydroxide formed externally on the rust spot.

The conditions of rusting of pure iron have never yet been studied, inasmuch as a pure iron in a massive form, if indeed in any form, is unknown.

DISCUSSION.

Dr. JOWETT said that he was interested to know that Prof. Tilden's experiments confirmed those carried out by himself many years ago. Although there were difficulties in the hydrogen peroxide theory of rusting, he would like to know how the electrolytic theory would account for the inhibiting effects of salts like sodium nitrite and potassium ferrocyanide, and the absence of this inhibiting effect by potassium nitrate and potassium ferricyanide, &c.

Prof. ARMSTRONG said that as they were carried out in his laboratory, he had enjoyed the opportunity of witnessing all Dr. Moody's experiments. One of his most striking observations was that a piece of iron, insulated from the glass by coating the ends with paraffin-wax, had remained unrusted after many months' exposure partly immersed in water and partly in air, but on turning the tube so that the metal came into contact with the glass at some point, the iron soon rusted. It therefore did not seem to be in a passive state. He (the speaker) thought the effect was perhaps due to silicic acid from the glass.

But even admitting, for the sake of argument, that treatment with chromic acid had rendered the iron passive, Prof. Tilden's results, important and valuable as they were, did not, in the speaker's opinion, in the least invalidate Dr. Moody's conclusions. The subject must be considered from the theoretical as well as from the practical side. Recalling the fact that, in 1885, in the course of the discussion on Mr. Brereton Baker's communication on "Combustion in Dried Gases," he had insisted that the conditions to be satisfied in the case of any chemical change were those involved in a galvanic cell—that the system must contain an electrolyte—Prof. Armstrong said that this contention had been entirely justified by later observations, especially by Mr. Baker's work on the interaction of hydrogen and oxygen. Not only was a dry mixture of these gases inert, but the presence even of liquid water was not sufficient to determine interaction; dirty water—water containing impurity which rendered it an electrolyte—alone conditioned the change. From this point of view, it was impossible that interaction should set in between iron, water, and oxygen; some trace of acid impurity must be present to condition the change.

Prof. TILDEN, in reply, recalled the familiar fact that water in contact with glass became alkaline and not acid, and that, although he could not profess to account for the effects produced by all the salts mentioned by Dr. Jowett, it was obvious that all those which, like alkalis, produced hydroxyl ions in aqueous solutions would tend to retard the process of rusting.

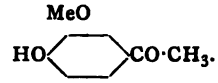
145. "*Studies on Zirconium.*" By EDGAR WEDEKIND and SAMUEL JUDD LEWIS.

The authors have studied the chemical and physical properties of zirconium. From a specimen containing 95·55 per cent of zirconium and 4·14 per cent of oxygen, the value 14 was obtained for the atomic volume and the values 6·078 and 6·753 for the atomic heat.

146. "*The Constituents of Canadian Hemp.* Part I. *Apocynin.*" By HORACE FINNEMORE.

The principal constituent of the root of *Apocynum cannabinum* (0·2 per cent) is identical with the crystalline apocynin of commerce, and with the product previously isolated by Wood (*Journ. Am. Med. Assoc.*, 1904, xliii., 1953) for pharmacological purposes.

Apocynin, $C_9H_{10}O_3$, which has a faint odour of vanillin, forms slender colourless prisms, m. p. 115°. It is convertible into a crystalline *potassium* derivative, and into *acetyl*, *benzoyl*, and *methyl* derivatives, m. p. 57°, 106°, and 51° respectively; it yields an *oxime* and a well-defined *hydrazone* and *semicarbazone*, m. p. 126° and 167° respectively; its molecule contains a single methoxyl group. As it has no aldehydic properties, it follows that apocynin is a hydroxymethoxyacetophenone. It is, in fact, identical with the acetovanillone (4-hydroxy-3-methoxyacetophenone) obtained by Tiemann (*Ber.*, 1891, xxiv., 2855), from *iso*eugenol, and by Goldschmiedt and Hemmelmayr (*Monatsh.*, 1894, xxxv., 338), from scoparin, and has the constitution (compare following abstract):—



The pharmacological properties of apocynin are under investigation. A synthesis of acetovanillone from vanillic acid by Neitzel (*Ber.*, 1891, xxiv., 2863) could not be confirmed, and another by T. Otto (*Ibid.*) gave a yield too small for practical use; the product, however, was identical with apocynin.

147. "*A New Synthesis of Apocynin.*" By HORACE FINNEMORE.

The previously known syntheses of apocynin (acetovanillone) being unsatisfactory (preceding abstract), the author has synthesised this substance from vanillin by an application of the Grignard process. Benzoylvanillin, $C_6H_3(OBz)(OMe)\cdot CHO$, is converted by magnesium methiodide, with subsequent hydrolysis into *benzoylapocynol*, $MeO\cdot C_6H_3(OBz)\cdot CH(OH)\cdot CH_3$, m. p. 128°, the phenolic benzoyl derivative of the secondary alcohol corresponding with apocynin. This benzoyl compound is oxidised by chromic acid mixture to *benzoylapocynin*, $MeO\cdot C_6H_3(OBz)\cdot CO\cdot CH_3$, m. p. 106°, which gives on hydrolysis 4-*hydroxy*-3-*methoxyacetophenone*, $MeO\cdot C_6H_3(OH)\cdot CO\cdot CH_3$, m. p. 115°; this is identical with natural apocynin, and with acetovanillone prepared from guaiacol (preceding abstract). The yields throughout are good.

Apocynol or 4-*hydroxy*-3-*methoxyphenylmethylcarbinol*, $MeO\cdot C_6H_3(OH)\cdot CH(OH)\cdot CH_3$, m. p. 101°, obtained by hydrolysing the above synthetic benzoyl derivative, may also be formed, although with poor yield, directly from apocynin by the action of sodium amalgam. This alcohol and the corresponding *dihydroxycarbinol*, $C_6H_3(OH)_2\cdot CH(OH)\cdot CH_3$, that is formed from it by the action of suitable hydrolytic agents, promise to be of interest pharmacologically by reason of their very close relation to adrenaline.

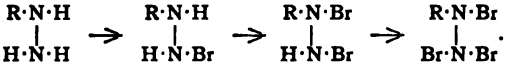
148. "*The Constitution of the Diazonium Perbromides.*" By FREDERICK DANIEL CHATTAWAY.

The view first suggested by Kekulé as an alternative and rejected by him, that the diazonium perbromides have the constitution $R\cdot NBr\cdot NBr_2$, that is, are tri-N-bromo-derivatives of the hydrazines, receives strong support from the fact that they are produced quantitatively when the required amounts of bromine are added to aromatic hydrazines dissolved in acetic acid.

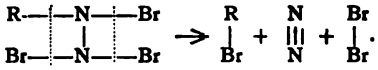
The author has recently shown (*Trans.*, 1898, xciii., 852) that diazonium bromides are produced quantitatively by the action of bromine on the hydrazines, and has expressed the view that these compounds when in acid solution are really di-N-bromo-substituted hydrazines

which are produced from the hydrazines by a process of substitution exactly similar to that which occurs when other compounds containing hydrogen attached to nitrogen are submitted to the action of bromine.

According to this view, the production of diazonium perbromides thus is the final and completing step in the replacement of the hydrogen of the $-NH\cdot NH_2$ group by bromine, thus :—



The entire behaviour of the diazonium perbromides is in accord with this view of their constitution ; for example, they are in exactly the condition necessary for the typical diazo-decomposition to take place; this, in fact, occurs when they are heated, suspended either in chloroform or glacial acetic acid, thus :—



All the members of the group of diazonium trihaloids have without doubt a similar constitution.

149. "*Cholestenone.*" By CHARLES DORÉE and JOHN ADDYMANN GARDNER.

Cholestenone, the ketone produced by the oxidation of cholesterol under suitable conditions, reacts with ozone in chloroform solution, giving an ozonide which most probably has the formula $C_{27}H_{44}O\cdot O_7$. This, when decomposed by water, gives carbon dioxide and a ketomonocarboxylic acid, $C_{26}H_{42}O_3$, which is identical with the one obtained by Windaus (*Ber.*, 1906, xxxix., 2008) by the oxidation of cholestenone with neutral permanganate. The work therefore confirms the opinion of Windaus that the double bond in cholestenone is situated in a terminal vinyl group. Cholesterol, on similar treatment, gives an ozonide, $C_{27}H_{46}O\cdot O_3$, which also evolves carbon dioxide on treatment with water.

Cholesterol therefore must possess a similar "carbon skeleton" to cholestenone, a point which had previously been regarded as doubtful.

150. "*Solubility of Silver Chloride in Mercuric Nitrate Solution.*" By BERTRAM HAWARD BUTTLE and JOHN THEODORE HEWITT.

The authors have studied the solubility of silver chloride in solutions of mercuric nitrate at 25°, using variable concentrations of mercuric nitrate, nitric acid, silver nitrate, and hydrochloric acid. Alteration in the concentration of the nitric acid, the other components remaining constant, has the effect of slightly increasing the precipitation of silver chloride.

From the results obtained when only the silver nitrate or hydrochloric acid is varied, the concentration of the other three components remaining unchanged, the authors arrive at the same conclusion as Morse (*Zeit. Phys. Chem.*, 1902, xli., 709), that when mercuric nitrate is present in large excess, chlorine only occurs as $HgCl\cdot$ ions.

151. "*The Relation between Absorption Spectra and Chemical Constitution.* Part IX. *The Nitroso- and Nitro-groups.*" By EDWARD CHARLES CYRIL BALY and CECIL HENRY DESCH.

The absorption spectra of compounds containing the —NO and the $-NO_2$ groups have been examined. In order to guard against the disturbing influence of the benzene nucleus, the majority of compounds examined were aliphatic, several inorganic compounds being included for the sake of comparison. The results obtained show that both groups give rise to definite selective absorption, the oscillation frequency of which, in the case of the —NO group, is strikingly low. Moreover, it is found that the nature of the atom to which the above groups are directly linked exerts a very considerable influence on the position of the band.

(To be continued).

FARADAY SOCIETY.

Annual General Meeting, June 23rd, 1908.

Prof. A. K. HUNTINGTON, Vice-President, in the Chair.

THE following Officers and Council were elected to serve for the ensuing year.

President—Sir Oliver Lodge, F.R.S.

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NOTICES OF BOOKS.

A Text-Book of Assaying. By C. and J. J. BERINGER. Eleventh Edition. London : Charles Griffin and Company, Limited. 1908.

THIS work is one which has stood the test of time, and has been found to meet the needs both of students and of analytical chemists. It has been kept thoroughly well up-to-date, and only comparatively small changes have been made in the eleventh edition. The chapter on tin now includes the wet method, and the results obtained by vanning and wet assays are carefully compared and discussed. No better text-book on general assaying is to be procured, and students in schools of mines and assayers should not fail to get a copy.

Chemical Reagents. Their Purity and Tests. By E. MERCK. Authorised Translation by HENRY SCHENCK, A.B. (Harvard). London : Archibald Constable and Co., Ltd. 1907.

THE issue of the report of the Committee on Purity of Reagents, appointed by the American Chemical Society in 1902, seems likely to be delayed for some years, since a considerable amount of work is still to be done, and a high standard of accuracy and precision is to be maintained. Hence the author of this book believes that in the meantime a revised translation of Merck's edition of Krauch's book "Die Prüfung der Chemischen Reagentien auf Reinheit" will be acceptable, and there is much truth in his contention that chemists will do wisely to make acceptance of their purchases conditional upon their coming up to the specifications of purity prescribed in this standard work. The text of the third edition of Krauch's book is closely followed, and the book contains the physical constants of all important and commonly used reagents, and full directions for testing them for purity. Some alterations and adaptations have been made for the benefit of English-speaking chemists, chiefly as regards the addition of acids of various strengths, *e.g.*, 99·5 per cent glacial acetic acid, which are to be obtained commercially in America but apparently not in Germany.

Laboratory Exercises in Physical Chemistry. By FREDERICK H. GETMAN, Ph.D. Second Edition. New York : John Wiley and Sons. London : Chapman and Hall, Limited. 1908.

AN elementary course of practical work in physical chemistry is given in this book, and the theoretical aspects of the subject are also discussed fully enough to enable the student to dispense almost entirely with reference to larger and more complete works until he has become fairly well

acquainted with the practical methods and results of physical chemistry. A chapter on thermostats has been added to the second edition, and also some notes on radio-active measurements, while the chapter on chemical dynamics has been extended. A good deal of the practical work described is to be regarded rather as belonging to the domain of pure physics than physical chemistry, and in some respects the author has not made the ideal choice of material. For instance, no experiments on osmotic pressure are included, and methods of molecular weight determination are not collected together, but are scattered throughout the book. Moreover, it might have been wiser to omit the short chapter on spectroscopic work, or else to enlarge it very considerably; in its present form students would probably find that it needs much supplementing from other works.

Post's Chemisch-Technische Analyse. (Post's Chemico-Technical Analysis"). Third Edition. Edited by Prof. Dr. BERNHARD NEUMANN. Vol. I., Part IV. Braunschweig: Friedrich Vieweg and Sohn. 1908.

WITH this number the issue of the first volume of the third edition of Post's "Text-book of Analytical Chemistry" is completed. It contains chapters on the inorganic acids, including hydrofluoric acid, and on potash, saltpetre, and other salts of potassium, and it has been thoroughly revised and much increased in size and value. The chapter on bromine has been re-written by Dr. Bokemüller. The last chapter, which is also new, deals with antichlors, sodium sulphide, alumina, &c. The index to the whole of the first volume is included in this part, and it is hoped that the second volume, of which two parts have already appeared, will be finished very shortly.

Decoration of Metal, Wood, Glass, &c. By H. C. STANDAGE. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1908.

THIS book contains a large collection of recipes for stains, dyes, &c., for use on metals, glass, wood, and porcelain. Methods of cleaning and renovating metals are described, and various miscellaneous processes, such as dyeing ivory, gilding book backs and edges, making imitation materials, etching, &c., are included. For painters and decorators and skilled workmen in the fancy trades the collection of formulæ will be of value, and the amateur may find in the book some suggestions which will save him the expense of employing outside assistance for small operations. It is certainly as useful as any book can be which deals with processes requiring in many cases considerable dexterity and long practice in order that they may be performed with anything approaching success.

Les Nouveautés Chimiques pour 1908. ("Chemical Novelties for 1908"). By CAMILLE POULENC. Paris: J. B. Baillière et Fils. 1908.

THE arrangement of this work is identical with that adopted in previous years. In the first chapter physical apparatus which is used more particularly in chemistry is described, such as Blackman's apparatus for determining vapour densities, Quidor and Nachet's microscope, which is specially adapted for stereoscopic micrography, and Pulfrich and Löwe's spectrograph for photographing spectra. The second chapter deals with new chemical apparatus, and contains details relating to François' oven, Schlemmer's distilling column, Forestier's gas generator, and similar contrivances due to many other workers. Chapter III. is devoted to electrical apparatus used in chemistry, one of the most interesting being Gasparini's arrangement for electrolytic oxidation. New analytical apparatus, burettes, pipettes, calorimeters, &c., are included in Chapter IV., while in the last chapter are grouped together all classes of apparatus used in bacteriology.

CORRESPONDENCE.

THE POTENTIAL ENERGY OF THE ELEMENTS.

To the Editor of the Chemical News.

SIR,—In reply to Mr. H. Stanley Redgrove in the CHEMICAL NEWS of July 3rd (vol. xcviii., p. 11) *re* my paper on "The Potential Energy of the Elements," I may mention that the values were obtained by me in the course of an investigation into the fundamental characters and correlation of the elements, on which I am still engaged. To explain the method of obtaining these values would necessitate entering into a much larger question which would require more space than I could expect you to afford me.—I am, &c.,

DANIEL J. RANKIN.

July 4, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Academie des Sciences. Vol. cxlvi., No. 23, June 9, 1908.

Direct Hydrogenation of Polyphenols. — Paul Sabatier and A. Mailhe.—The general method of direct hydrogenation by contact with divided nickel can be applied to the transformation of benzene and its homologues into cyclohexane and its homologues. It can also be used to convert phenols into the corresponding cyclohexanols, aniline and its substituted derivatives into cyclohexylamine and its homologues. The method as originally described cannot, however, be applied to di- and triphenols, which under the action of nickel above 200° yield benzene, phenol, cyclohexanol, and cyclohexane. By lowering the temperature of hydrogenation considerably it is possible to fix six atoms of hydrogen on to hydroquinone. pyrocatechin, and pyrogallol. The temperature is kept at 130°, and a very rapid current of hydrogen is passed over the polyphenol in presence of reduced nickel.

Catalytic Dehydration of Organic Compounds.—J. B. Senderens.—Precipitated alumina rendered anhydrous by drying below a red heat is an excellent catalysing agent, excelling silica, magnesium phosphates, &c., in this respect. (When too strongly heated alumina is a bad catalyser). Ethyl oxide yields ethylene, and from acetic acid acetone can readily be obtained. Precipitated alumina is also capable of removing one molecule of hydracid; thus isobutylchloride at 250° yields hydrochloric acid and isobutylene.

Action of Silver Nitrate on Chloroauric Acid and Preparation of Fulminating Gold.—Jules Jacobsen.—When silver nitrate is added to a solution of chloroauric acid a precipitate containing all the gold and all the chlorine is obtained:—

$$AuCl_3HCl + 4AgNO_3 + 3H_2O = Au(OH)_3.4AgCl + 4HNO_3.$$

This precipitate gives with ammonia a gelatinous precipitate of fulminating gold, the silver chloride being dissolved out. The product possesses high explosive power. It is more stable when wet than when dry. The formula is $Au(OH)_2NH_2$ or $(AuN.2H_2O)$.

Separation of Ammonia and Amines by means of Boiling Absolute Alcohol.—Jean Bertheaume.—The author has studied the solubility in absolute alcohol of monomethylamine chloride, ammonium chloride, and a mixture of these two substances, and has found that when a mixture is treated with boiling absolute alcohol, filtered, and the filtrate evaporated, a minimum of 8·5 per cent of ammonium chloride is retained by the amine.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xli., No. 9, 1908.

Simple Test for Glycuronic Acid by means of Naphthoresorcin, Hydrochloric Acid, and Ether.—B. Tollens.—With hydrochloric acid and phloroglucin arabinose, xylose, and glycuronic acid all give colour and spectral reactions, but when heated with hydrochloric acid and naphthoresorcin the two pentoses give a strong fluorescence but no characteristic colour or spectral reactions, while glycuronic acid differs from them in giving a blue coloration and a spectral band on the D line. The blue substance is soluble in ether, to which it imparts a blue or blue-violet colour, and its formation, together with the appearance of the dark spectral band lying near the D line, can be employed as a sensitive test for glycuronic acid.

Detection of Nickel and Cobalt when present together.—Hermann Grossman and Walter Heilborn.—Ammonia is added to the fairly concentrated solution containing the two metals, and then a 10 per cent solution of cane-sugar and dicyandiamidin-sulphate solution (Grossmann's "nickel reagent"). Finally caustic soda is added. When much nickel is present a yellow or reddish-yellow coloration is produced, while the presence of large quantities of cobalt causes the solution to turn red or reddish-violet. Thus fractions of a milligrm. of cobalt in presence of more than 100 times the quantity of nickel can be detected by the violet coloration, while if much cobalt and only a little nickel is present, crystalline nickel dicyandiamidin is always precipitated. This test is very accurate and rapid.

Picrates.—W. Suida.—Picric acid and anthranilic acid form two compounds containing one molecule of picric acid to one molecule of anthranilic acid, and one molecule of picric acid to two molecules of anthranilic acid respectively. This behaviour of anthranilic acid is not due to its amphoteric character, for similar well characterised compounds of toluidine with picric acid are formed. Both compounds in which the molecular proportions are 1 : 1 are faintly coloured, while those containing one molecule of picric acid to two molecules of base are strongly coloured, both when hydrated and in the anhydrous state.

Solutions of Alkali Metals in Liquid Ammonia.—Otto Ruff and Julian Zedner.—The authors have confirmed their conclusions that the substances described by Joannis as metallic ammonium compounds are really the pure metals with a deposit of supersaturated solution in liquid ammonia upon them. For this purpose they have plotted the boiling and melting-point curves of ammonia solutions of potassium, sodium, and lithium of very different concentrations. From the melting-point diagram, especially with sodium, it is seen that within a certain interval of temperature two liquid phases exist between fairly wide concentration limits. It seems not improbable that a compound of formula $Na.5NH_3$ exists.

Hydrogen Persulphide.—Ignaz Bloch and Fritz Höhn.—Hydrogen persulphides corresponding to the formulæ H_2S_2, H_2S_3, H_2S_5, H_2S_6, H_2S_7, and H_2S_8 have been prepared, though in no case in a pure state. The best characterised of these are the derivatives of the compounds H_2S_6 and H_2S_8, which have been obtained and analysed by different investigators. By the action of dilute hydrochloric acid upon sodium polysulphide at a temperature from −10° to about 0°, an oil consisting of raw hydrogen persulphide can be obtained. From the analysis of the purified product it is clear that Rebs' statement, that whatever polysulphide is used the compound formed is always H_2S_5, is incorrect. Unless special precautions are taken no single chemical compound is obtained.

Hydrotrisulphide.—Ignaz Bloch and Fritz Höhn.—Raw hydrogen persulphide prepared from sodium polysulphide when subjected to fractional distillation in an apparatus through which a current of dry hydrochloric acid gas has been led (to neutralise the alkalinity of the glass) yields a distillate which has the composition H_2S_3. Hydrotrisulphide is a colourless liquid with a sharp smell, recalling carbon disulphide and camphor. When warmed it yields H_2S. It is inflammable and is decomposed by alkalis. Concentrated sulphuric acid reacts slowly with it, giving sulphur dioxide. It behaves like a reducing and sulphating agent, and is also capable of acting as an oxidiser.

Hydrodisulphide.—Ignaz Bloch and Fritz Höhn.—When the receiver in which the hydrotrisulphide described in the preceding article is cooled, it is found that the percentage of H_2S in the product is high; this is due to the presence of some disulphide, H_2S_2. When the receiver is gently heated and the product is received in another cooled vessel, a yellowish, clear, liquid consisting of pure H_2S_2 is obtained. It is inflammable, and is decomposed by distilled water and by alkalis. Concentrated sulphuric acid also decomposes it, yielding amorphous sulphur and sulphur dioxide.

Constitution of Hydrodisulphide and Hydrotrisulphide.—Ignaz Bloch.—The compounds of hydrogen and sulphur appear to be formed by successive substitutions of HS for H in the lowest member of the series H_2S. Thus $HS.H \rightarrow HS.HS$ or H_2S_2; $HS.HS \rightarrow HS.S.HS$ or H_2S_3. Undoubtedly higher members of this series exist. Corresponding polysulphides are known, *e.g.*, $(NH_4)_2S$, $(NH_4)_2S_2$, $(NH_4)_2S_3$, $(NH_4)_2S_4$, $(NH_4)_2S_5$, $(NH_4)_2S_7$, $(NH_4)_2S_8$, $(NH_4)_2S_9$.

MISCELLANEOUS.

The Seventh International Congress of Applied Chemistry will be held in London, from May 27th to June 2nd, 1909. An Organising Committee has been formed for the purpose of making all arrangements for the holding of the Congress in London. This Committee consists of representatives of the following Societies:—Royal Societies of London, Edinburgh, and Dublin, Society of Chemical Industry, Chemical Society, Institute of Chemistry, Society of Public Analysts, Royal Society of Arts, Iron and Steel Institute, Institution of Mining Engineers, Institution of Mining and Metallurgy, Society of Dyers and Colourists, International Association of Leather Chemists, Institute of Brewing, Royal Agricultural Society of England, The Lawes Agricultural Trust, Pharmaceutical Society, Royal Photographic Society, Faraday Society, London Chamber of Commerce (Chemical Trade Section), representing all the most important industries. Previous Congresses have been held in Brussels (1894), Paris (1896), Vienna (1898), Paris (1900), Berlin (1903), and Rome (1906). The work of the Congress has steadily grown in importance, and it is hoped that the London Congress may be a worthy successor to those which have gone before. The scope of the work of the Congress is indicated by the following list of Sections and Presidents:—Analytical Chemistry—Dr. T. E. Thorpe, C.B., F.R.S. Inorganic Chemistry and Allied Industries—Dr. Ludwig Mond, F.R.S. Metallurgy and Mining—Sir Hugh Bell, Bart. Explosives—Sir Andrew Noble, Bart., K.C.B., F.R.S. Organic Products—Prof. W. H. Perkin, F.R.S. Colouring Substances and their Uses—Prof. Meldola, F.R.S. Industry and Chemistry of Sugar—Richard Garton, Esq. Starch Industry—Dr. Horace T. Brown, F.R.S. Fermentation—John Gretton, Esq., M.P. Agricultural Chemistry—Lord Blyth. Hygiene and Medical Chemistry—Sir J. Crichton-Browne, F.R.S. Pharmaceutical Chemistry—N. H. Martin, Esq. Bromatology—Robert R. Tatlock, Esq., F.I.C. Photographic Chemistry—Sir W. de W. Abney, K.C.B., F.R.S. Electrical and Physical Chemistry—Sir John Brunner, M.P. Law, Political Economics, and Legislation with reference to Chemical Industries—Lord Alverstone. Committees are being formed in the different countries for the purpose of furthering the interests of the Congress. At a later date, further particulars will be sent out; meantime, the undersigned will be happy to give any explanations that may be desired—Wm. Macnab, Honorary Secretary, 10, Cromwell Crescent, S.W.

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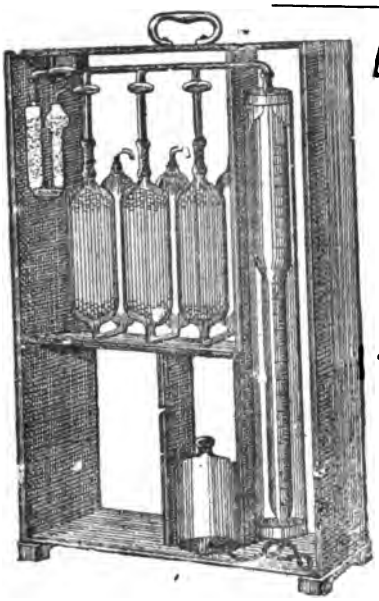

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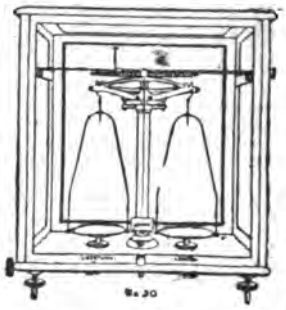

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THE CHEMICAL NEWS.

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ON THERMO-CHEMICAL EVIDENCE FOR VON BAEYER'S STRAIN THEORY.

By H. STANLEY REDGROVE, B.Sc. (Lond.), F.C.S.

Introduction.

It has already been pointed out that the thermal behaviour of the hydrocarbons whose molecular heats of combustion have been determined by Thomsen are in agreement with Baeyer's strain theory (see Chemical News, xcv., 301). The argument is repeated here at greater length in order that the following new calculations may be perfectly intelligible.

Considering, firstly, the "ethene" and "ethine" links. If an ethene link were a double link in an arithmetical sense we should have $L_2 - 2L_1 = 0$, whereas actually $L_2 - 2L_1 = 2(2H - L_1) - (4H - L_2) = 2\beta - \gamma = 16{\cdot}0$ cals. Similarly, $L_3 - 3L_1 = 3(2H - L_1) - (6H - L_3) = 3\beta - \delta = 44{\cdot}5$ cals. We know, however, from pure chemistry that a "double" link is not an arithmetical double link, and that a "treble" link is not an arithmetical treble link, but in so far as stability is concerned each is less than a "single" link.

The instability of ethane and ethine links finds a mathematical interpretation in von Baeyer's strain theory, which states that the four valencies of the carbon atom normally act in the directions of the four lines drawn from the centre to the four corners of a regular tetrahedron (*i.e.*, making an angle of 109° 28′ with one another). If the directions of the valencies are different from this normal condition a state of intramolecular strain is set up, the magnitude of the strain depending upon the angle through which the valencies are diverted.

The tendency of the strain is to sever the link, hence the instability of such links; moreover, the molecular heat of combustion of a compound, which is in a condition of intramolecular strain, will be greater than the calculated by the thermal equivalent of this strain. Hence, if we express this thermal equivalent for the "double" link by T_2, and for the "treble" link by T_3, then $2L_1 + T_2 = L_2$, whence $T_2 = 16{\cdot}0$ cals., and $3L_1 + T_3 = L_3$, whence $T_3 = 44{\cdot}5$ cals.

In the polymethylenes, although there are no "double" or "treble" links, we can readily see by constructing models of the molecules that they are in a condition of strain. Assuming that they possess a regular configuration and that the atoms constituting the ring are all in one plane, we obtain for the angle of deviation the values shown in Table I., the general formula being:—

$$D^\circ = 54^\circ\,44' - \frac{n-2}{n}\,90^\circ,$$

where n is the number of carbon atoms in the ring and D° the angle of deviation.

Table I.

Ring.	D°.
Dimethylene (=ethylene)	54° 44′
Trimethylene..	24° 44′
Tetramethylene	9° 44′
Pentamethylene	0° 44′
Hexamethylene	−5° 16′
also—	
Acetylene	70° 32′

This explains to a great extent the relative stabilities of the various groups of polymethylenes.

The molecular heat of combustion of trimethylene determined by Thomsen (*i.e.*, 498·0 cals.) was found to be considerably greater than the calculated (*i.e.*, 474·9 cals.). This excess (which we will represent by $T_{III.}$) is consequently the thermal equivalent of the intramolecular strain in this compound, so that $T_{III.} = (498{\cdot}0 - 474{\cdot}9)$ cals. = 23·1 cals.

Thomsen's determinations (save that of the molecular heat of combustion of ethylene oxide) do not afford further data for testing the accuracy of von Baeyer's strain theory. However, Stohmann has carried out a research on the heats of combustion of polymethylene compounds. The

Table II.

		M.H.C. constant volume (Stohmann). Cals.	Mean. Cals.
1. Dimethylenes (ethylenes)*—			
Fumaric acid..	$C_4H_4O_4$	320·7	320·7
Succinic acid..	$C_4H_6O_4$	357·1	357·1
Therefore $\beta - T_2 = 36{\cdot}4$ cals.			
Dimethyl fumarate ..	$C_6H_8O_4$	664·7	664·7
Dimethyl succinate ..	$C_6H_{10}O_4$	703·3	703·3
Therefore $\beta - T_2 = 38{\cdot}6$ cals.			
Mean $\beta - T_2 = 37{\cdot}5$ cals.			
2. Trimethylenes—			
aa-Trimethylenedicarboxylic acid..	$C_5H_6O_4$	483·5	483·95
aβ-Trimethylenedicarboxylic acid..	$C_5H_6O_4$	484·4	
Glutaric acid..	$C_5H_8O_4$	515·0	515·85
Methyl succinic acid ..	$C_5H_8O_4$	515·2	
Ethyl malonic acid ..	$C_5H_8O_4$	517·9	
Dimethyl malonic acid .	$C_5H_8O_4$	515·3	
Therefore $\beta - T_{III.} = 31{\cdot}9$ cals.			
aoββ-Tetramethyl trimethylenetetracarboxylate	$C_{11}H_{14}O_8$	1170·7	1170·7
Tetramethyl methylenedimalonate	$C_{11}H_{16}O_8$	1202·2	1202·2
Therefore $\beta - T_{III.} = 31{\cdot}5$ cals.			
Mean $\beta - T_{III.} = 31{\cdot}7$ cals.			
3. Tetramethylenes—			
aa - Tetramethylenedicarboxylic acid	$C_6H_8O_4$	642·4	642·45
aβ - Tetramethylenedicarboxylic acid	$C_6H_8O_4$	642·5	
Adipic acid	$C_6H_{10}O_4$	668·6	671·84
Methylglutaric acid ..	$C_6H_{10}O_4$	670·5	
Ethyl succinic acid ..	$C_6H_{10}O_4$	671·9	
Sym. dimethyl succinic acid	$C_6H_{10}O_4$	670·7	
Unsym. dimethyl succinic acid	$C_6H_{10}O_4$	671·4	
Propyl malonic acid ..	$C_6H_{10}O_4$	674·7	
*iso*Propyl malonic acid .	$C_6H_{10}O_4$	674·9	
Methyl ethyl malonic acid	$C_6H_{10}O_4$	672·0	
Therefore $\beta - T_{IV.} = 29{\cdot}4$ cals.			
4. Pentamethylenes—			
aβ - Pentamethylenedicarboxylic acid	$C_7H_{10}O_4$	775·7	775·7
Pimelic acid	$C_7H_{12}O_4$	828·3	828·3
Therefore $\beta - T_{V.} = 52{\cdot}6$ cals.			
5. Hexamethylenes—			
cis-Hexahydrotere-phthalic acid	$C_8H_{12}O_4$	928·0	928·45
trans - Hexahydroterephthalic acid	$C_8H_{12}O_4$	928·9	
Suberic acid	$C_8H_{14}O_4$	982·8	982·8
Therefore $\beta - T_{VI.} = 54{\cdot}35$			

* Maleïc acid is omitted as it gives a divergent result.

first part of this paper deals with the interpretation of Stohmann's experimental results, the second with ethylene oxide.

1. *Interpretation of Stohmann's Experimental Results for the Molecular Heats of Combustion of Polymethylene Compounds.**

The procedure adopted by Stohmann and his collaborators was to determine the heat of combustion of polymethylene carboxylic acids and the corresponding open-chain acids.

The difference between the molecular heats of combustion of any polymethylene compound and the corresponding open-chain compound, is due to the breaking of the ring and the addition of two hydrogen atoms linked to carbon; in terms of our constants this difference equals $2H - L_1 - T$; *i.e.*, $\beta - T$.

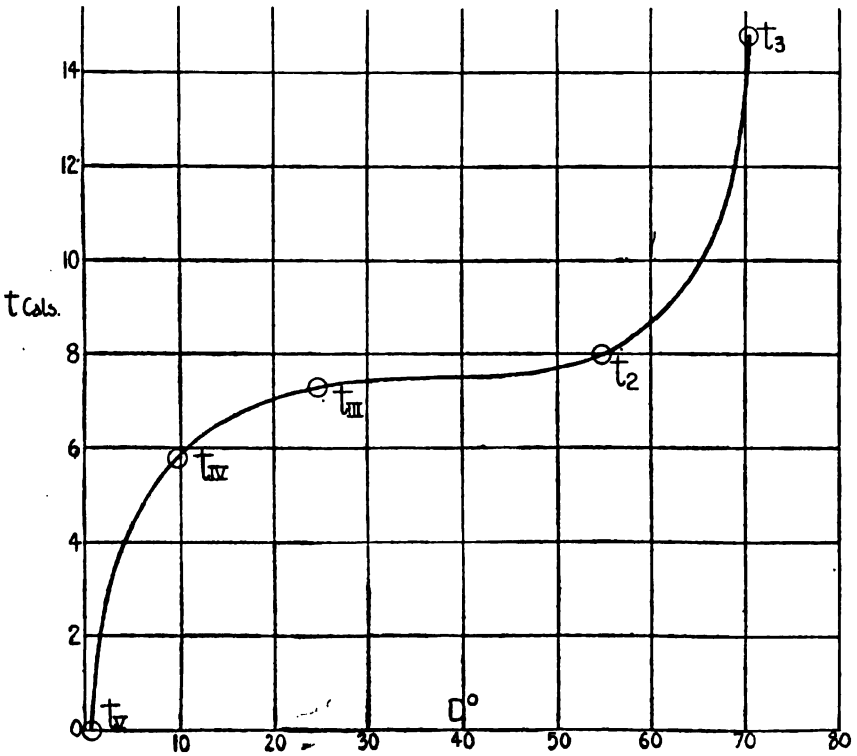
only 1·0 cal. in the case of T_2 and 2·3 cals. in the case of T_{III}.

TABLE III.

Ring.	Stohmann. Cals	Cals.	Thomsen. Cals.
Acetylene . .	—		$T_3 = 44·5$
Ethylene	$T_2 = 52·5 - 37·5$	= 15·0	$T_2 = 16·0$
Trimethylene . .	$T_{III.} = 52·5 - 31·7$	= 20·8	$T_{III.} = 23·1$
Tetramethylene	$T_{IV.} = 52·5 - 29·4$	= 23·1	
Pentamethylene	$T_{V.} = 52·5 - 52·6$	= −0·1*	
Hexamethylene	$T_{VI.} = 52·5 - 54·35$	= −1·85*	

* Zero.

Now, as in the above rings varying numbers of links are concerned, it is evident that, in order to obtain comparable results, it will be necessary to divide the thermal equivalent



In Table II. are given these molecular heats of combustion (for constant volume), and the values of $\beta - T$ calculated for each ring. The notation employed is to denote the number of carbon atoms in the ring by a Roman numeral affixed to T.

The value of β being known, the values of T can be at once obtained, as in Table 3. Perfectly accurate results cannot be expected, for the value of β (= 52·5 cals.) is calculated from the molecular heats of combustion of gases, whereas the values of $\beta - T$ are for solids. However, as $\beta - T$ is always found as the *difference* between two of Stohmann's experimental results, the error introduced will be at a minimum, and fairly good approximate results may be hoped for.

On comparing with the above those values of T which have also been found entirely from Thomsen's experiments (namely, T_2 and $T_{III.}$) we see the difference amounts to

* See Stohmann, Kleber, and Langbein, *Journ. Prakt. Chem.*, xl., 202; Stohmann and Kleber, *Ibid.*, xliii., 1, and xlv., 475.

of the total strain in each ring (T) by the number of links in that ring which are in a condition of strain, thereby obtaining the thermal equivalents of the strain set up by deviating one C.C link through various angles, represented by t in Table 4.

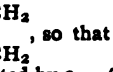
TABLE IV.

Ring.	Thermal equivalent of strain for one link. Stohmann. Cals.	Thomsen. Cals.	Mean. Cals.	Angle of deviation. D°.
Acetylene . . t_3 =	—	14·8	—	70° 32′
Ethylene . . . . t_2 =	7·5	8·0	7·75	54° 44′
Trimethylene . . $t_{III.}$ =	6·9	7·7	7·3	24° 44′
Tetramethylene $t_{IV.}$ =	5·8			9° 44′
Pentamethylene $t_{V.}$ =	0·0			0° 44′

It appears from this table that the value of t depends upon that of D° (although the relation is evidently not that of a simple proportion), for as D° increases, t increases. This regularity is best exhibited by a curve obtained by

plotting D° and t along co-ordinate axes (the values used being Thomsen's for t_3 and t_2; Stohmann's for $t_{IV.}$ and $t_{V.}$; and the mean of Thomsen's and Stohmann's for $t_{III.}$). Its regular nature is at once apparent, but up to the present the author has not been able to satisfactorily represent the relation between D° and t by any simple formula.

2. *Interpretation of Thomsen's Experimental Result for the Molecular Heat of Combustion of Ethylene Oxide.*

Chemical evidence shows that ethylene oxide has the constitution  , so that its molecular heat of combustion is represented by $2a-\beta-\omega$.* The molecular heat of combustion (corrected for constant volume) of this compound, was found by Thomsen to be 311·7 cals., whereas the calculated amounts to only $(2\times210{\cdot}8-52{\cdot}5-75{\cdot}4)$ cals. = 293·7 cals., so that the experimental is considerably greater (by 18·0 cals.) than the calculated molecular heat of combustion. The molecular heat of formation deduced from the experimental molecular heat of combustion, namely, 17·2 cals., is correspondingly less than the calculated, namely, $2a'-\beta'-\omega' = (2\times21{\cdot}2-15{\cdot}0+7{\cdot}8)$ cals. = 35·2 cals.

This apparently anomalous result would, however, be expected from the standpoint of von Baeyer's strain theory, for from analogy with trimethylene the ethylene oxide molecule must be in a state of strain. The thermal equivalent of this strain is now given as 18·0 cals.; that it is not identical with that for trimethylene is only to be expected from the fact that carbon has an affinity for carbon differing from that which it has for oxygen, and, moreover, it is not improbable that the normal directions of the oxygen valencies are different from the normal directions of carbon valencies.

The conclusion to be drawn from the thermal data considered, is that it is in perfect accord with von Baeyer's strain theory, thereby supplying satisfactory evidence of the general validity of this theory.

The Polytechnic, Regent Street, W.

EXPLOSIVE CRYSTALLISATION.

By FRANK E. WESTON, B.Sc.

In the course of an investigation that is being carried out in this laboratory it was found necessary to attempt the crystallisation of a solution in a partial vacuum. The solution was an aqueous one of a compound formed by the combination of a sulphate with a thiosulphate. The solution, contained in a 6 in. evaporating basin, was placed on a porcelain dish partially filled with concentrated sulphuric acid, the latter dish standing on a ground glass plate, ¼ in. thick by 12 in. square; the whole was covered with a bell-jar, 7 in. diameter and 8 in. high, fitted with a cork carrying a glass tap, and made perfectly air-tight by sealing the surface of contact of bell-jar and plate with paraffin wax. The solution was allowed to evaporate for three weeks at a pressure of about half an atmosphere, during which time it evaporated very slowly. At the end of this period the pressure was reduced by means of a water-pump to about 20 mm., and was so left on Wednesday evening, July 1st, at 9.30 p.m. On Thursday morning, July 2nd, at 10 a.m., on going to examine the solution, it was found that the basin was broken into several pieces, the solution was splashed all over the bell-jar, the glass plate cracked into a dozen pieces, and a large star-shaped crack was found in the shoulder of the bell-jar; the bell-jar was still firmly fixed to the glass plate and the solution had partially percolated through the cracked plate into the wooden base; on opening the tap and carefully removing the jar it was found to be full of SO_2, evolved by the action of H_2SO_4 on the thiosulphate. The apparatus had been touched by no one, and consequently no reason could be assigned for what had happened. On Friday, July 3rd, an attempt was made to crystallise two solutions of a sulphite *in vacuo*. The solutions were saturated (and probably supersaturated) and were placed in a similar apparatus excepting that fused $CaCl_2$ took the place of the H_2SO_4, and the second basin rested on a tripod stand that stood on the glass plate. The pump was set working and in half-an-hour the pressure had fallen to about 20 mm.; suddenly the top basin was seen to bump violently, and its contents partly ejected with a dull thud, and before the apparatus could be touched the basin had broken into half-a-dozen pieces, which on falling disturbed the bottom basin and upset its contents; at the same time the whole apparatus seemed to be shaken, with the result that the glass plate was cracked into several pieces and air violently entered the jar.

The bell-jar still remained fimly fixed to the plate, and on removing it no trace of SO_2 was evident, nor had any apparent decomposition taken place. Some of the pieces of the broken basin had a fine crystalline deposit on them.

It seems probable that crystallisation was suddenly induced on the surface of the basin, with a consequent sudden increase in the vapour pressure of the surrounding liquid, which, under the very low pressure existing in the bell-jar, caused the liquid to boil violently, the reacting force causing the basin to bump violently and hence to break; the phenomenon was accompanied with a slight but distinct explosive noise.

Further experiments are being carried out with a view to confirming the theory put forward, and other solutions which are known to be supersaturated are also being examined.

The Chemical Laboratory,
The Polytechnic, Regent Street, W.,
July 4th, 1908.

* For the meaning of ω see CHEMICAL NEWS, xcvii., p. 253.

THE HOMOGENEITY OF TELLURIUM.

By VICTOR LENHER.

When tellurium or its dioxide reacts with a number of the liquid anhydrous chlorides, crystals of tellurium tetrachloride or of a double chloride are formed along with a mother-liquor which contains the excess of the reacting anhydrous chloride (see CHEMICAL NEWS, xcviii., p. 4). This mother-liquor contains such by-products of the reaction as may be soluble in it, and has also been found invariably to contain greater or less quantities of tellurium.

When sulphur monochloride is the reacting liquid, the amount of tellurium appearing in the excess of the reagent is quite small, so small indeed that a fairly good separation of tellurium from selenium can be effected by use of this reagent in the cold, inasmuch as selenium dissolves readily in sulphur monochloride. When, however, the sulphur chloride is warm, its solvent power on tellurium chloride is materially increased.

Other chlorides studied, such as thionyl chloride, sulphuryl chloride, phosphorus oxychloride, and antimony pentachloride, when in excess, dissolve considerable quantities of tellurium chloride or of the double chloride as the case may be.

Inasmuch as the question of the complexity of tellurium has been repeatedly raised, especially so after the prediction of Mendeleeff in his London address (*Journ. Chem. Soc.*, lv., 649), when he predicted that tellurium contained another element, whose atomic weight would be 212, and which he called dvi-tellurium; and after the work of Brauner (*Ibid.*, lv., 411), when he thought from some of his experiments that the so-called element was actually a composite, it has been deemed important to examine these mother-liquors, arising from the reactions above indicated, to ascertain whether any of the reagents studied would possibly allow of a splitting apart of tellurium.

Norris, Fay, and Edgerley (*Am. Chem. Journ.*, xxiii., 105) studied the elementary character of tellurium by carrying out an elaborate fractional crystallisation of the double bromide of tellurium and potassium. Later Norris (*Journ. Am. Chem. Soc.*, xxviii., 1675) made fractional distillations of tellurium oxide, following the distillations by determinations of the atomic weight. The conclusions drawn by Norris are that his results have furnished positive evidence in favour of the elementary character of tellurium.

Baker and Bennet have recently published an account of their investigations on the atomic weight and elementary character of tellurium (*Journ. Chem. Soc.*, xci., 1849). Their work was carried on for a period of thirteen years. They studied the fractional crystallisation of telluric acid, the progressive solubility of barium tellurate, the fractional distillation of the metal, chloride and dioxide, the fractional decomposition of the hydride, the fractional electrolysis of the bromide and chloride, and the fractional precipitation of the chloride by water. They have been unable to distinguish any difference in the atomic weight, and consider 127·6 to be the true atomic weight.

Study of the Products of the Reaction of various Anhydrous Chlorides on Tellurium and its Oxide.—The mother-liquor arising from each of the reactions studied was decomposed by water, and the tellurium precipitated from hydrochloric acid solution by sulphur dioxide, after which it was carefully purified from such materials as might be introduced by the reagents used.

In each case the tellurium obtained was found to answer to all of the tests to which the original material responded, and behaved in all respects similarly to what we commonly know to be tellurium.

The method of approaching this problem can be illustrated by the experiments in which the action of phosphorus oxychloride on tellurium dioxide was studied. In this case a crystalline compound is found which careful analysis has shown to have the composition $TeCl_4.POCl_3$. The excess of phosphorus oxychloride used was observed to carry considerable quantities of tellurium in solution. This mother-liquor was carefully decanted from the crystallised salt and treated with water. By this procedure, the phosphorus oxychloride is converted into phosphoric and hydrochloric acids and the tellurium tetrachloride decomposed into hydrated dioxide.

After addition of sufficient hydrochloric acid to completely dissolve the tellurium dioxide, treatment with sulphur dioxide yielded elementary tellurium. It has been observed in working with this particular liquid that when an attempt is made to precipitate tellurium out of a solution containing a large quantity of phosphoric acid, the reaction is very much retarded and the tellurium precipitates very slowly. This can be overcome, in a large measure, by the addition of hydrochloric acid and considerable dilution with water. The retardation of precipitation of tellurium in presence of phosphoric acid is doubtless due to lack of dissociation, and the precipitation can better be effected after addition of considerable quantities of hydrochloric acid.

The tellurium, after having been converted into chloride by means of aqua regia, was again precipitated by sulphur dioxide, converted into the basic nitrate, and crystallised from nitric acid solution, as has been suggested by Norris, Fay, and Edgerley for the purification of tellurium (*Am. Chem. Soc.*, xxiii., 105). The basic nitrate, when ignited, gave the dioxide.

Determination of the amount of tellurium in the dioxide by precipitation as element gave the ratio—

TeO_2	0·85635
Te	0·6845
Atomic weight Te..	127·52

The tellurium thus obtained from the excess of phosphorus oxychloride showed by the ratio of tellurium dioxide to element that no apparent change in atomic weight had aken place, and that no splitting apart of the element had occurred was evidenced by the fact that the salt $TeCl_4.POCl_3$ which did not dissolve in the excess of phosphorus oxychloride, and which contained all of the tellurium not appearing in the mother-liquor, showed by analysis a rational formula with tellurium taken at the commonly accepted atomic weight.

The mother-liquors appearing in all the reactions of the anhydrous chlorides which were brought in contact with tellurium or the dioxide were examined in a manner similar to that with phosphorus oxychloride, and the same character of results were obtained. A rational formula for the insoluble crystalline product always appeared when a careful analysis was made, while the tellurium appearing in the mother-liquor, after decomposing the excess of reagent and purifying the element from such products of the reaction as might contaminate it, always showed the ratio of tellurium dioxide to element corresponding to the atomic weight of 127·5 for tellurium.

Tellurium and Ferric Chloride.—By heating together in the dry condition tellurium and anhydrous ferric chloride, reaction ensues with the production of ferrous chloride and tellurium tetrachloride. This reaction can also be accomplished and much more conveniently so by the continued action of a hot solution of ferric chloride on tellurium, $4FeCl_3 + Te = TeCl_4 + 4FeCl_2$. Pethybridge (U.S. Patent No. 709,037, Sept. 16, 1902) has studied the action of a solution of ferric chloride having a density of 1·18 on telluride gold ores, and claims to be able to extract the tellurium as tetrachloride with the simultaneous formation of ferrous chloride, leaving behind gold and silver as follows:— $8FeCl_3 + (AuAg)Te_2 = 2TeCl_4 + 8FeCl_2 + (AuAg)$.

This is in line with the results previously obtained by the author (*Journ. Am. Chem. Soc.*, xxiv., 355), which indicate that the tellurium in the natural tellurides of gold is not strongly united to the gold.

While tellurium reduces ferric chloride to ferrous with the simultaneous production of the tetrachloride, ferrous salts will precipitate a part of the tellurium when added in excess to a solution of the tetrachloride in hydrochloric acid.

Crane (*Am. Chem. Journ.*, xxiii., 408) and Rose (*Pogg. Ann.*, xxi., 443) have attributed this precipitation of small quantities of tellurium by means of ferrous sulphate to the presence of tellurium dichloride in the tetrachloride. Tellurium dichloride is, however, decomposed by water or aqueous hydrochloric acid into the element and oxide or chloride, and with the knowledge that tellurium dissolves in hydrochloric acid in the presence of the air to tetrachloride directly, it would seem that the precipitation of small quantities of tellurium by means of ferrous salts is largely controlled by mass action. The reactions between ferric chloride and tellurium and ferrous salts and hydrochloric acid solutions of the tetrachloride are indeed reversible.

In view of this partial precipitation of tellurium by ferrous salts, a series of experiments was conducted, in which this partial precipitation was made the basis of a fractionation. To this end a quantity of tellurium originally obtained from the Baltimore Smelting and Rolling Co. was precipitated from a caustic soda solution by means of grape-sugar. The carefully washed material was dissolved in aqua regia and was precipitated by sulphur dioxide from a hydrochloric acid solution. The tellurium thus obtained was fused with pure potassium cyanide, the resulting fusion extracted with water, and a current of air passed through the solution to precipitate the tellurium out of the solution of the alkaline telluride, leaving sulphur or selenium present in solution as alkaline thiocyanate or selenocyanate. The tellurium purified in this manner was introduced into a porcelain boat which was placed in a porcelain tube. This tube was connected with a supply of hydrogen, which was generated from pure zinc and hydrochloric acid and which was washed with water. The tellurium was distilled from the boat (the tube being heated by a blast lamp) into the cooler portion of the tube where it was condensed. The distilled metal was collected and

introduced again into a clean porcelain boat and tube and the distillation repeated.

Twenty-five grms. of this metal were dissolved in a mixture of nitric and hydrochloric acids and a hydrochloric acid solution of the tetrachloride formed by boiling off the excess of nitric acid. To this solution was added a solution containing about 25 grms. of ferrous sulphate, which contained a small amount of ferric salt, when a precipitate of about 0·1 grm. of elementary tellurium appeared. The quantity of precipitate obtained did not appear to be materially increased by the addition of a considerably larger quantity of the ferrous sulphate solution. The solution containing the tellurium and iron salts was treated with acid sodium sulphite and the tellurium precipitated. This tellurium was then re-converted into chloride by means of hydrochloric and nitric acids, and ferrous sulphate again added to the hydrochloric acid solution. A second precipitate of about the same size as before appeared. This operation was repeated twenty-four times, fractions being obtained each time of approximately the same size. These twenty-four precipitates were combined, dissolved in hydrochloric and nitric acids, and a hydrochloric acid solution obtained as before. Treatment of this solution with ferrous sulphate again yielded a small precipitate of tellurium, the size of the precipitate being about the same as in the previous series of fractions. A second precipitation was made and was combined with the previous one for an atomic weight determination. The metal was treated with nitric acid in a small Jena flask, and the resulting basic nitrate gradually heated to 440°, when the weight was found to be constant. 0·1694 Te gave 0·2119 TeO_2, and if O = 16, Te = 127·55.

Inasmuch as the original material showed the same atomic weight, it is evident that by this method of fractionation no portion can be found which shows a figure radically different from the commonly accepted figure.

Action of Hydrochloric Acid on Tellurium in presence of Air.—Although tellurium is ordinarily considered as insoluble in hydrochloric acid, yet it is actually attacked slightly by the acid when exposed to the air, if sufficient time is allowed for contact. This action can be demonstrated by bubbling a current of air, for several weeks, through concentrated hydrochloric acid in which is suspended metallic tellurium. In a comparatively short time the acid becomes yellow, indicating the presence of tetrachloride.

This action of hydrochloric acid on tellurium in the presence of air has been made the basis of an experiment in which it would be possible to have differential solution take place should tellurium contain a higher member of the series of elements in which it is commonly placed.

One hundred grms. of carefully purified tellurium was precipitated from hydrochloric acid solution by means of sulphur dioxide, and the precipitate washed until the wash-water would not react for chloride with silver nitrate. This freshly precipitated and finely divided metal was introduced with pure concentrated hydrochloric acid into a filtering flask, and a current of air which had been previously washed with water and then passed through pure hydrochloric acid was drawn through the solution for three months. At the end of this time a considerable part of the metal had dissolved. The solution was filtered, and on treatment of the solution with acid sodium sulphite, tellurium was precipitated. Two portions of tellurium were here obtained, one of which was a smaller portion of a few grms. obtained from a tellurium solution that had been formed by the slow action of hydrochloric acid and air on tellurium, and this solution had remained in contact with the main part of the finely divided metal for three months. The portion which dissolved and that still remaining undissolved were found to be identical in chemical character, obeyed the same reactions, and when converted into oxide, and this oxide analysed, gave an atomic weight of 127·5.

In the hands of the author, tellurium has shown no signs of breaking apart in any of the reactions which had for their object a study of the elementary character of the metal, nor in the various reactions or derivatives which have been studied in our laboratory has the element shown any indications of other than simple character.

The statements of Mendeleeff as to the possibility of tellurium containing another element, and his views as to its incorrect atomic weight, have led to an extended series of critical studies on this subject. Brauner studied the atomic weight for six years, Norris for nine, Baker and Bennet for thirteen, and the author for ten. We see, therefore, thirty-eight years of experimental work directed to the study of the atomic weight of an element which to-day remains as an element whose elementary character is well established, and which stands with a higher atomic weight than the next element in the horizontal series. While it still remains an exception to the periodic arrangement of Mendeleeff, and so far as its comparison with iodine is concerned, is abnormal, yet in the main points of its chemical behaviour and those of its compounds, it properly belongs associated with sulphur and selenium in the sixth group.

The element tellurium still remains, however, an exception to the periodic arrangement of Mendeleeff, its atomic weight being higher than that of iodine. We must hence conclude that either tellurium is abnormal in a direction which has not yet received careful study, or that we do not yet appreciate all of the principles of the periodicity of the elements. — *Journal of the American Chemical Society*, vol. xxx., No. 5.

RECENT DEVELOPMENTS OF THE KJELLIN AND ROCHLING-RODENHAUSER ELECTRIC INDUCTION FURNACES.*

By J. HARDÉN.

THE object of this paper is to discuss the rapid development of the electric induction furnace during the last two years. Before proceeding to discuss the practical improvements in detail, it will be interesting to touch upon one or two novel points of a purely physical nature. We will consider one which will be of general interest.

The "Pinch" Effect of an Electric Current.

Many people experimenting with electric arc and resistance furnaces have observed that when a current is passed through a bath of molten metal or electrolyte the current density cannot be raised to a very high limit without distorting the bath. If the current density is raised to a certain point, it is plainly seen that the metal carrying the current contracts violently, or, in other words, becomes pinched, the effect of which is that the metal on either side of the pinch swells up above the average level of the bath. If the current is still increased, the pinch effect becomes so complete as to actually cause separation in the bath, thereby breaking the current circuit and causing a violent flash, together with rapid volatilisation. This is only a momentary effect, as the separate parts immediately flow together again, closing the circuit, when the same phenomenon repeats itself. Thus it was found impossible to pass an unlimited amount of power into the bath, although one might be well below the boiling-point of the metal. The explanation of this pinching is quite obvious. We have only to remember that conductors carrying current in the same direction have a mutual attraction for each other, and if we consider that the molten bath consists of an infinite number of elementary paths of current, it is natural that, as each one is attracting the paths in its immediate neighbourhood, there must be a tendency to compress the full section of the bath. If now, therefore, the bath is slightly contracted at any one spot, the current density will here be raised and the pressure

* A Paper read before the Faraday Society, June 23rd, 1908.

will increase until finally the bath becomes so pinched that an actual break in the circuit may take place. Of course this is only momentary.

Correspondents of the technical papers have discussed this question, and the point has been raised whether this phenomenon can also be observed in the induction furnace. Theoretically it is obvious that it should, for although the induced current is an alternating one, it has for a given moment of time the same direction in each part of the path.

We have taken observations under working conditions in a 60-kw. Kjellin furnace erected in London for experimental work. A small charge of pig-iron, consisting of about one-third of the full capacity of the furnace, was placed in the bath, and a current of 20 kw. was employed. As soon as the charge was fully liquid the pinching effect commenced, and the metal was seen to contract at a certain spot, raising the level of the metal on both sides of the pinch, which was sufficient to break the circuit, causing a flash, immediately after which the metal flowed together again, closing the circuit. The level of the metal immediately on each side of the pinched area rose about 1½ in. above the normal level of the bath. It was found, on examination, that a small piece of slag was burnt into the bottom of the hearth at the point where this phenomenon occurred, thus causing the original reduction of area. Pieces of pig-iron were added to the bath, and as the depth increased the pinching slowly disappeared. It was found that if the original charge was about half of the full charge no pinching effect could be observed.

Another striking feature is the resistance curve of the melt—that is to say, the curve obtained by taking the voltage across the terminals of the furnace during melting. Supposing we are starting the charge by meant of a cold ring of welded or cast-iron. Putting on a full load and keeping the kilowatts constant, we first find that the voltage rises above the normal for full load, and as the ring gets hotter the voltage still gradually rises until the ring becomes a bright red heat. From that point a decided drop in the voltage is noticed, although the power is kept constant and the weight of the charge is kept the same. As soon as the ring begins to melt the voltage again rises, but does not reach the same value as before the ring was red-hot. Now let us consider the cause of this result. This furnace is, as you know, nothing but a transformer with a short-circuited secondary. This latter must be placed some distance from the primary, owing to the thickness of the lining, cooling chamber, &c. We therefore have a certain amount of magnetic leakage, not only around the primary, but also round the secondary. The secondary is of iron, with comparatively high permeability, and we have therefore introduced an easy path for the lines of the stray field, hence the increased inductive voltage across the terminals. This is to a certain extent compensated for by the lower ohmic resistance of the ring; but as the resistive coefficient of the latter is such as to increase with the temperature, the total resistance, measured across the terminals, will increase with the temperature, but the power factor will be lower during this period, which shows that it is not only the ohmic resistance that is increased, but also the inductive resistance. This is due to the fact that the permeability of the iron is also increased to a certain extent with the temperature; in fact, it rises very rapidly up to a temperature of about 840° C., when the permeability begins to drop very quickly, and reaches zero at about 920° C. In this interval between 840° and 920° the inductive resistance is rapidly decreasing, because the easy path for the stray field is checked, and the voltage across the terminals is consequently lowered. But in the meantime the ohmic resistance is steadily increasing, but more slowly than the change in the permeability; therefore the voltage will again rise, though slowly, until the temperature is reached at which the loss by radiation and the heat introduced balance each other. It may even increase somewhat above this point, owing to oxidation of the charge, but this increase is very slight. This is the explanation why some people were misled into believing that the increase of resistance in iron due to heat was not a straight line curve. (It may, perhaps, not be so, but the effect shown on the furnace terminal is certainly produced in the way stated—which can be proved by the wattmeter readings).

Now, turning our attention to the more practical side of our process, we recognise a new feature, which is likely to prove a distinct improvement, viz., the Röchling-Rodenhauser modification of the induction furnace.

In the original Kjellin furnace some disadvantages are experienced when dealing with material which has to be refined and treated in very large quantities. For instance, when a charge of three tons or more is to be treated, the section of the bath becomes very large, thus causing a low resistance, whereby the power factor is lowered. If we try to increase the resistance by making the ring wider in diameter and of smaller section, the distance from the primary will be greater and the power factor again lower. Thus it becomes necessary to employ a generator of very low periodicity for such furnaces, which is, of course, undesirable. Also the processes of desulphurisation and dephosphorisation are very tedious, as it is difficult to keep the slag sufficiently liquid for such purposes. Nevertheless, this class of furnace will still hold its own, as it forms an almost ideal crucible steel furnace. In the case of crucible steel we have seldom to deal with more than one and a-half to two tons at a time, and as it does not pay to use impure raw material, no refining is required, but plain melting and "killing," and for this class of work plain induction furnaces can be provided, which will answer very well at 15 to 25 cycles per second. But when it is desired to refine, say, a material smelted from inferior ores and decarburised in a converter, but still containing up to 0·1 to 0·2 per cent cent sulphur and 0·05 or more phosphorus, in quantities of five to seven tons, this plain induction furnace would not be so satisfactory.

This was what was required for making rail and other similar steel at the Röchling'sche Iron and Steel Works at Völklingen, Germany, and therefore the engineers at these works, Mr. Rodenhauser and Dr. Schönawa, set themselves to adapt the Kjellin furnace, and so arrived at the "combined furnace." This consists of a transformer furnace with two ring-shaped baths adjacent and communicating with one another, in the case of a single-phase furnace, and three such baths in the case of a three-phase furnace, with a square or rectangular hearth in the centre between the rings, with doors in front and behind, in exterior appearance very much like a Siemens open-hearth furnace; but the principal feature is a heavy secondary winding of copper cables, placed around and conaxial to the primary (one on each leg of the core), surrounded by the rings forming the charge. These copper secondaries, consisting of a few turns only, are connected to conductive plates—they can hardly be called electrodes, for reasons given below—built into the furnace wall, two in front and two at the back for a single-phase furnace. These plates consist of corrugated cast steel plates, and a compound of magnesite, dolomite, and tar is applied firmly over the corrugation. The plates do not conduct well when cold, but as soon as the furnace is charged with molten raw material they will act as a "conductor of the second class," and readily allow the current to pass. Thus about one-half of the power is transmitted to the charge by induction in the rings and the rest of the power through the side plates. As the copper secondary is placed very close to the primary, the leakfield is very much smaller; in fact, three furnaces for one to one and a-half tons are now in operation with 50 periods at a power factor of 0·7 to 0·85, a result which could never be obtained with a plain induction furnace of a similar size, in spite of "bifilar" baths and other devices which have been tried. But *this* is not the chief advantage, as the same result may be obtained by other electrical means. A far more important gain is to be found in the metallurgical possibilities obtained with the new design. We know that for carrying out any

refining process in steel we need a sufficiently liquid slag, and ways and means of handling the same. This is to a certain extent obtained in some "electrode furnaces," where an arc plays between carbon blocks and the slag "blanket." This, however, in some cases, has proved troublesome, the drawback being that the temperature must be extremely and unnecessarily high at the spot where the arcs are playing, which is not without certain disadvantages to some steels. If we try to dip the carbons direct into the molten metal, we find that they are consumed at once, in such case contaminating the steel, which is, of course, difficult to avoid.

But this is not so in the case of the combined furnace. The conducting side plates before mentioned are of quite a neutral nature; in fact, some plates, which have been in constant use day and night for three months, were so little corroded at the end of that period that the loss of the plates, calculated per ton of steel, was hardly determinable. Part of the power is induced in the rings, thus heating the charge, and the rest passes through the side plates, to such an extent only as experience has proved to be necessary in order to obtain a sufficiently liquid slag. The ring-shaped part of the bath is covered with bricks, at a height below the level of the charge in the centre bath. Thus no slag can enter into the rings, and as it is the slag which is injurious to the lining, the rings need hardly any repair during a long run, whereas the rectangular bath in the middle is easily accessible, and can easily be patched out.

The lining is simply burnt magnesite or dolomite, mixed with tar, and stamped in hot.

It has been said that the use of these steel side plates would be equal to a return to the old system of electrodes, with all their disadvantages, but it is evident from what has been stated above that this is not so, as practically no consumption whatever of these plates takes place, and they can hardly be called "electrodes" in that sense of the word.

Let us now follow up the progress of the operation. After the lining is stamped in, the tar is burnt out (either by heating a cast steel ring or pouring a small quantity of pig-iron into the hearth), leaving behind a sintered mass, forming a solid brick of basic lining. The pig-iron is teemed for treatment in the Bessemer converter, and a fresh charge is given, which is tapped direct from the converter. It is more economical to burn out the carbon and the silicon in the converter before refining from phosphorus and sulphur. The larger furnace at Völklingen will take a charge of four tons.

Calcined lime is added to form a suitable slag; this slag also contains about 6 per cent of magnesia. Sometimes a small quantity of fluor-spar is also added to act as a flux, but this is not always necessary. Plate scale from the rolling-mill is added for decarburising.

In this condition the slag will take up the phosphorus very readily, after which it is made more viscous by applying cold lime, and drawn off through the slag door by a slight tilting of the furnace.

It is essential for a successful dephosphorisation that the charge should be what is called "hot brittle"—*i.e.*, have an excess of oxygen, in order to prevent the phosphorus wandering back into the charge again. After removing the slag which contains phosphorus, ferrosilicon or carbon is added, forming SiO_2 or CO, thus depriving the charge of the oxygen. It has been found that the adding of ferrosilicon will shorten the time of the deoxidation; thus, if power is cheap the cheaper carbon may be employed, and in the case of dearer power it is better to use the ferrosilicon.

As soon as the dephosphorising is effected this first slag is entirely removed, and a fresh slag of lime only is formed, which, when the temperature is raised, acts as a desulphuriser in forming iron sulphide. The oxygen is also driven out in this operation, partly by forming calcium carbide, and partly by adding a small quantity of ferrosilicon, and eventually some mill scale.

After this, the maximum power is given in order to drive out the last trace of oxygen, and as soon as no more gas bubbles are seen to leave the charge a test piece is taken out and forged. If too soft for the purpose, some coke powder is thrown in until the right proportions are arrived at. As a rule, the charge is finished in one and a quarter to two hours, but if necessary the steel can, without disadvantage, be kept in the furnace for ten hours or more. It is thus possible to treat a material which contains up to 0·1 per cent phosphorus and 0·1 per cent sulphur or more, so that a sulphur product containing 0·006 per cent phosphorus and 0·02 per cent sulphur, and from 0·5 to 0·1 per cent manganese and 0·01 silicon, will be obtained.

As to the power consumption, if the furnace is charged with molten material from the converter, the consumption is from 125 to 150 kw. hours per ton of finished material. This, of course, depends upon the quality of the raw material, but 130 kw. hours may be taken as a good average.

The finished product is especially distinguished by its great strength, equality, and homogeneity. In fact, rails have been made with a much higher bending and breaking point than ordinary Bessemer or Thomas rails, and these rails command from 25s. to 45s. per ton more than ordinary rails, owing to their greater durability. There is no necessity to give a large quantity of figures as to strength, &c., of this material, but a few may be useful.

Carbon	0·075
Phosphorus	0·03
Manganese	0·594
Sulphur	0·069
Silicon	—

Breaking sixty-four tons per square cm., elongation 21 per cent, contraction 28·5 per cent.

Carbon	0·086
Phosphorus	0·018
Manganese	0·310
Sulphur	0·077

Breaking 37·3 per cent, elongation 36·5 per cent, contraction 69·2 per cent.

Discussion.

Mr. F. W. HARBORD, while holding the modified furnace to be a decided improvement on the original, doubted whether this type of electric furnace could compete against the modern converter or open hearth furnace for making rail steel. It was essentially a crucible furnace of large size. He was interested to note that CaC_2 was formed *in situ* in the furnace.

Dr. J. A. HARKER said they hoped at the National Physical Laboratory to make determinations of some of the urgently needed physical constants of metals at high temperatures in the small experimental Kjellin furnace which had been presented to the laboratory. This type of furnace, in which metals could be melted in a neutral atmosphere, was ideal for the purpose. The new metallic filament lamps were greatly in need of a vacuum induction furnace.

Mr. E. RISTORI remarked that it was necessary to fall back on the resistance principle as soon as the induction furnace was required to refine.

Alkaline Chloroiridates and Chloroiridites.—Marcel Delépine.—The chloroiridates of potassium, rubidium, cæsium, and ammonium can be obtained by double decomposition with the sodium compound, $IrCl_6Na_2+6H_2O$. Their general formula is $IrCl_6M_2$. The chloroiridites of sodium, potassium, and ammonium can be prepared by reducing the corresponding iridates in suspension, or dissolved in water by means of neutral oxalate, and warming on the water-bath. $2IrCl_6M_2+C_2O_4M_2=2IrCl_6M_3+2CO_2$. The salts of the type $IrCl_6M_3$ readily lose water, while the dimetallic salts, $IrCl_5(H_2O)M_2$, suffer no loss at 150°, and do not change colour; the water they contain is water of constitution.—*Comptes Rendus*, cxlvi., No. 24.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

Part III.—Analysis of the Aluminium and Iron Groups, including Beryllium, Uranium, Vanadium, Titanium, Zirconium, and Thallium.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 17).

General Discussion (continued).

12. The filtrate from the chloric acid precipitate is first tested for phosphate. When it is not present, the iron, thallium, titanium, and zirconium are separated from the other elements by the addition of ammonium hydroxide. When phosphate is present, in order to separate it from the alkaline earth elements, the basic acetic precipitation is employed, ferric chloride being first added if necessary. The provision here made for the case that phosphate is present is thought to have many advantages over the methods ordinarily employed in schemes of qualitative analysis, where the phosphate is removed by tin in nitric acid solution, by ferric chloride and barium carbonate, or by ferric chloride and ammonium acetate in the first stages of the analysis of the group. Of these three processes, the basic acetate is much more rapid and simple of execution; but it does not give a separation which is at all satisfactory when applied to a solution containing all the elements of the aluminium and iron groups; thus chromium and zinc may in certain combinations of elements be found either in the precipitate or filtrate, and manganese also divides, unless great care is taken to make the precipitation in a large volume at the proper acid concentration. In the scheme of analysis here presented, this basic acetate separation has been introduced only after these troublesome elements have already been removed, for the presence of phosphate involves no complications in the preceding steps of the process. Under these circumstances it is no more difficult to secure accurate results in this separation than in the precipitation with ammonium hydroxide. Indeed, by adopting this process for all cases the complication of the alternative procedure and the special test for phosphate might be removed; but since the operations require a somewhat longer time, it has seemed best to retain the ammonium hydroxide precipitation for the case that phosphate is not present.

13. The iron might be separated from the titanium and zirconium by the well-known method of boiling a solution of the elements kept slightly acid with sulphurous acid; but it is difficult to secure complete precipitation of the titanium and zirconium and at the same time to prevent the carrying down of iron. Besides this, the operation is a long one, involves large dilution, and makes it necessary to use hydrofluoric acid in re-dissolving the precipitated hydroxides. On the other hand, the removal of the iron by the method of Rothe (*Stahl und Eisen*, 1892, xii., 1052; 1893, xiii., 333), which consists in shaking it out of a strong hydrochloric acid solution by means of ether, is extremely simple and rapid, gives a perfect separation, and leaves the titanium and zirconium in solution. We have therefore unhesitatingly employed this method in our scheme of analysis. Moreover, in this process the thallium, which is present as thallic chloride, is extracted together with the ferric chloride by the ether; it can be readily detected, after evaporating off the ether and reducing the ferric and thallic salts with sulphurous acid, by the formation with potassium iodide of the characteristic yellow precipitate of thallous iodide.

14. For the separation of the titanium and zirconium from each other we have adopted the process of Hillebrandt (*Bull. U.S. Geol. Survey*, 1900, No. 176, p. 75), which consists in adding sodium phosphate to a slightly acid solution containing hydrogen peroxide, whereby the zirconium is precipitated and the titanium remains in solution. The latter is shown to be present by the colour of the solution, but it can also be precipitated as phosphate by destroying the hydrogen peroxide with sulphurous acid after filtering off the zirconium compound.

15. The analysis of the ammoniacal solution containing nickel, cobalt, and perhaps zinc and the alkaline-earth elements, is carried out along the conventional lines. The first three elements are precipitated by the addition of ammonium sulphide, and the zinc is extracted, if the precipitate is large, by treating it with cold dilute hydrochloric acid, or if it is small by dissolving the precipitate completely and treating again with sodium peroxide. Instead of separating the nickel and cobalt from each other, it was found to be shorter and more conclusive to divide the solution of the sulphides into two parts and to test one portion for nickel by adding potassium cyanide and sodium hypobromite and the other portion for cobalt with potassium nitrite. The conditions for securing the best results in the nickel test were fully studied. Finally, in the filtrate from the ammonium sulphide precipitate the alkaline-earth elements are precipitated with ammonium carbonate as usual.

16. Though no provision is made in the system of analysis for the separate detection of any of the rare-earth elements, yet a process has been described for determining whether any of them are present and for removing them when they are found to be in the solution. This process consists in evaporating the acid solution of the original ammonium sulphide precipitate, adding to the residue hydrofluoric acid, and filtering. This converts the rare-earth elements completely into insoluble fluorides, and enables them to be separated from all the other elements of the aluminium and iron groups (except from a very large quantity of aluminium). When alkaline-earth elements are present, these are also precipitated as fluorides, wholly or in part, and are separated from the rare-earth elements by decomposing the fluorides with sulphuric acid, diluting, filtering off any akaline earth sulphates that separate, and precipitating the rare-earths in the filtrate with ammonium hydroxide. This process of isolating the rare elements has been worked out in the Research Laboratory of Physical Chemistry of the Massachusetts Institute of Technology by W. C. Arsem and H. I. Wood, for use as a group separation in the "System of Qualitative Analysis including nearly all the Metallic Elements" (two parts of this System have already been published—see *Technology Quarterly*, 1903, xvi., 93, and 1904, xvii., 214; Chemical News, 1906, xciii., p. 134 *et seq.*).

17. Final confirmatory tests are given for almost all the elements; and much experimenting has been done on some of these tests in order to make them delicate and reliable; thus, this is true of the colour tests for aluminium and zinc made by igniting the oxides with cobalt nitrate, of that for chromium with hydrogen peroxide and ether, of that for nickel with H_2S in an alkaline tartrate solution, and of that for vanadium made by adding H_2O_2 to an acid solution; also of the precipitation test for uranium with potassium ferrocyanide. No satisfactory tests have as yet been found for beryllium or zirconium. Many, but not all, of these confirmatory tests will be found superfluous and will be omitted by the experienced analyst, except in cases where a very small precipitate or one of doubtful character is obtained; but they will, we believe, be useful to those unfamiliar with this scheme, and they serve, in the case of students, the educational purpose of making them acquainted with additional reactions of the elements in question.

18. It may be thought an objection to this scheme of analysis that it involves a number of manipulative operations unusual in qualitative analysis, such as heating in a closed bottle, saturating the acid-ether solution with hydrogen chloride gas (in the separation of aluminium and beryllium), filtering through an asbestos filter (in the separation of manganese), and shaking out with ether in the separation of iron from titanium and zirconium.

* From the *Journal of the American Chemical Society*, xxx., No. 4.

These operations, when they have been once executed, are found to be little if any more troublesome than the ordinary operations of precipitation and filtration. They are, moreover, mostly employed only in connection with the detection of the rarer elements, where the difficulties in finding any satisfactory method are so great that a little additional trouble is an insignificant factor. And finally, from an educational standpoint, they introduce the student to new kinds of processes, thus enlarging his knowledge and diminishing the force of the objection that the ordinary study of qualitative analysis is too limited in its scope.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 18th, 1908.

Sir William Ramsay, K.C.B., F.R.S., President, in the Chair.

(Concluded from p. 22).

152. "*Benzeneazo-2-pyridone.*" By William Hobson Mills and Sibyl T. Widdows.

An alkaline solution of 2-pyridone couples with benzenediazonium chloride to yield bright yellow 5-*benzeneazo*-2-*pyridone* (m. p. 210—212°), the constitution of which has been established by the following synthesis:—6-*Chloronicotinamide* (m. p. 210—211°) was converted by sodium hypobromite into 2-*chloro*-5-*aminopyridine* (m. p. 82—83·5°). This reacts with nitrosobenzene to form 2-*chloro*-5-*benzeneazopyridine* (m. p. 108—109°) which was converted by sodium methoxide into 5-benzeneazo-2-pyridone, identical with the above product.

3-Hydroxypyridine also couples in alkaline solution with benzenediazonium chloride, giving rise to *benzeneazo*-3-*hydroxypyridine* (m. p. 167—169°).

The preparation of 5-*amino*-2-*pyridone*, its characterisation by its *dibenzoate*, $C_6H_5{\cdot}CO{\cdot}NH{\cdot}C_5H_3N{\cdot}O{\cdot}CO{\cdot}C_6H_5$ (m. p. 211—212°), and its production together with aniline by reduction of 5-benzeneazo-2-pyridone, were also described. The base was prepared in the two following ways:—1. From hydroxynicotinic acid by Curtius' method (*Ber.*, 1894, xxvii., 778); ethyl hydroxynicotinate (m. p. 149—150°) is transformed through the *hydrazide* (m. p. 310°) and *azide* (m. p. 139—140°) into *bishydroxypyridylcarbamide*, $CO(NH{\cdot}C_5H_3N{\cdot}OH)_2$, which gives rise to the base on hydrolysis. 2. From the above 2-chloro-5-aminopyridine by the action of sodium methoxide.

153. "*The Electrolytic Chlorination of the Salts of some Organic Acids.*" By John Kenneth Harold Inglis and Fred Wootton.

The authors have examined the products obtained by the electrolysis of solutions containing a mixture of potassium halide with the potassium salt of an organic acid. It was found necessary to protect the liquid from the reducing action of the hydrogen by means of a porous pot. The results varied with the temperature and type of electrode employed. In the case of a solution containing potassium benzenesulphonate and potassium chloride, to which most attention has so far been paid, tri- and tetra-chloro-*p*-benzoquinone were formed if the solution was kept hot and a platinum-foil electrode used. At the same time, evidence was obtained of the production of mono-, di-, and trichlorobenzenesulphonates, substitution in the nucleus having taken place. These substitution products have not yet been satisfactorily separated. In a similar manner, using potassium bromide, tetrabromo-*p*-benzoquinone was produced. A platinum-gauze electrode gave different products, which have not yet been investigated. Indefinite results were obtained with sodium benzoate, phenol, and *p*-nitrophenol. When iodide was used instead of chloride, no substitution was detected.

154. "*The Action of Nitrous Gases on Dicyclopentadiene.*" By Alexander Rule.

The author has investigated the crystalline products formed by the action of nitrous gases on a well-cooled ethereal solution of di*cyclo*pentadiene.

The gaseous products from the action of nitric acid on arsenious oxide bring about the formation of a mixture which, on separation by means of alcohol, was found to consist of the ψ-nitrosite and the dinitro-derivative of the hydrocarbon.

The ψ-nitrosite is insoluble in cold alcohol, but on boiling is gradually dissolved, being at the same time decomposed into two molecules of nitro*iso*nitrosodi*cyclo*pentadiene. Sodium methoxide acts on the ψ-nitrosite with the formation of *iso*nitromethoxydi*cyclo*pentadiene and sodium hyponitrite. The methoxy-derivative, on treatment with nitric acid, forms a brilliant blue ψ-nitrol, which is very unstable.

The dinitro-derivative is soluble in alcohol, and thus may be easily separated from the ψ-nitrosite. It forms characteristic crystals, which melt at 121°.

Sodium hydroxide acts on the dinitro-derivative, forming *nitrohydroxydi*cyclo*pentadiene*, which also forms a characteristic unstable blue *ψ-nitrol* with nitrous acid. The *sodium* salt of nitrohydroxydi*cyclo*pentadiene is obtained as a crystalline mass by cooling the alkaline solution, but the free hydroxy-compound shows very little tendency to crystallise.

155. "*An Alternative Structure for the supposed Stereoisomeric α-Osazones.*" By Frederick Daniel Chattaway.

By adding iodine to an ethereal solution of benzaldehydephenylhydrazone containing sodium ethoxide, Ingle and Mann (*Trans.*, 1895, lxvii., 606) obtained two isomeric substances of the formula $C_{26}H_{22}N_4$, melting at 186° and 208° respectively. The isomeride of lower melting-point was identified with diphenyldibenzylidenehydrotetrazone, first obtained by Minunni (*Gazetta*, 1892, xxii., [2], 217) by oxidising benzaldehydephenylhydrazone with mercuric oxide, and subsequently by von Pechmann (*Ber.*, 1893, xxvi., 1045), using amyl nitrite as the oxidising agent.

Ingle and Mann made the further observation that the second isomeride, melting at 208°, was transformed into ordinary benzilosazone on heating with various solvents. Entirely on the ground that stereoisomerides of benzilosazone were required by the Hantzsch-Werner hypothesis, they assumed that their newly discovered compound was one of these much sought for isomerides, and named it benzil-α-osazone. In order to determine its configuration, they heated both it and ordinary benzilosazone with cold concentrated sulphuric acid, and found that from the former only were they able to obtain, among other products, benzaldehyde. From this circumstance, they favoured the *syn*-configuration for the supposed new modification.

Biltz (*Annalen*, 1899, cccv., 165; cccviii., 1) discovered later that Ingle and Mann's benzil-α-osazone and a whole series of similar compounds could be obtained by dissolving the corresponding hydrazones in alcohol in presence of potassium hydroxide and oxidising them by a current of air. He observed that, like the first discovered compound, all these so-called α-modifications could be transformed by heat into the corresponding β-modifications derivable directly from the diketones. Biltz offers no evidence that these new compounds are stereoisomerides of the ordinary osazones, but accepts Ingle and Mann's assumption that this is the case; indeed, he seems to regard both isomerides as formed by direct removal of hydrogen attached to carbon, and formulates the reaction thus:—

$$\begin{array}{l} C_6H_5{\cdot}C{:}N{\cdot}NH{\cdot}C_6H_5 \\ \quad | \\ \quad H \\ \quad H \\ \quad | \\ C_6H_5{\cdot}C{:}N{\cdot}NH{\cdot}C_6H_5 \end{array} + O = \begin{array}{l} C_6H_5{\cdot}C{:}N{\cdot}NH{\cdot}C_6H_5 \\ \quad \| \\ C_6H_5{\cdot}C{:}N{\cdot}NH{\cdot}C_6H_5 \end{array} + H_2O.$$

Ingle and Mann in their paper record an observation which, properly interpreted, gives a far more likely explanation of this isomerism than that afforded by the Hantzsch-Werner hypothesis, and in this connection it should be noted that in no case has the third modification of these osazones predicted by this hypothesis been obtained. They show that the diphenyldibenzylidenehydrotetrazone obtained as one of the products of their reaction undergoes intramolecular re-arrangement into ordinary benzil-β-osazone on heating with alcoholic potash.

Now, this isomeric change of diphenyldibenzylidene-hydrazotetrazone into benzilosazone under the influence of alcoholic potash must without doubt take place in two stages, thus :—

$$C_6H_5\cdot N\cdot N:CH\cdot C_6H_5 \qquad C_6H_5\cdot NH\cdot N:C\cdot C_6H_5$$
$$\Big| \rightarrow \qquad \Big| \rightarrow$$
$$C_6H_5\cdot N\cdot N:CH\cdot C_6H_5 \qquad C_6H_5\cdot N\cdot N:CH\cdot C_6H_5$$

$$C_6H_5\cdot NH\cdot N:C\cdot C_6H_5$$
$$\rightarrow \Big|$$
$$C_6H_5\cdot NH\cdot N:C\cdot C_6H_5,$$

and, in the absence of all evidence to the contrary, Ingle and Mann's benzil-α-osazone (m. p. 208°) ought certainly to be regarded as the intermediate product of this transformation. The presence of benzaldehyde among the products of its decomposition by sulphuric acid is direct evidence in favour of this view. The author has recently shown that hydrogen attached to nitrogen in aromatic hydrazines is attacked with extraordinary readiness by free oxygen (Chattaway, *Trans.*, 1897, xci., 1323), and his observations leave little doubt that the primary products when hydrazones are oxidised by air are hydrotetrazones, which thus undergo more or less complete transformation according to conditions. In Biltz's experiments, when the temperature is low, the first stage of transformation only occurs, the so-called α-osazones, which are really compounds of the general formula $\begin{matrix} R\cdot NH\cdot N:C\cdot R \\ | \\ R\cdot N\cdot N:CHR \end{matrix}$, being formed; on the other hand, when the temperature is higher, the second stage of the transformation occurs, and the β-compounds result, these only being true osazones of the general formula $\begin{matrix} R\cdot NH\cdot N:C\cdot R \\ | \\ R\cdot NH\cdot N:C\cdot R \end{matrix}$.

Baly, Tuck, Marsden, and Gazdar (*Trans.*, 1907, xci., 1579) have recently examined the absorption spectra of benzilosazone and its so-called α-isomeride. Finding them identical, they regard this as very striking and clear proof that the substances have the same configuration in solution. This appears to be a wholly unwarranted conclusion. Although such band absorptions, when different, may point to a difference of constitution, it can hardly be claimed that similarity of absorptions in such complex molecules as those in question can prove identity of configuration. To do this it would first be necessary to show that in presence of four phenyl groups a shift of one linking from nitrogen to carbon would produce an appreciable effect.

156. "*The Formation of 4-Pyrone Compounds from Acetylenic Acids.*" (Part II.). By SIEGFRIED RUHEMANN.

2-Phenyl-6-methyl-4-pyrone, which is formed by the action of sodium ethoxide on a mixture of acetone and ethyl phenylpropiolate (*Trans.*, 1908, xciii., 431), has been examined on lines similar to those which have been followed up by Feist (*Annalen*, 1890, cclvii., 253) in his work on dimethyl-4-pyrone. Under the influence of baryta, the former compound is transformed into *benzoylacetylacetone*, $C_6H_5\cdot CO\cdot CH_2\cdot CO\cdot CH_2\cdot CO\cdot CH_3$ (m. p. 106—107°). This triketone does not condense with aldehydes in the presence of piperidine or alkalis as catalysts. It reacts with two molecules of phenylhydrazine to yield a colourless *substance*, $C_{24}H_{22}N_4$ (m. p. 174—175°), which only slightly reduces Fehling's solution on boiling, and which, most probably, has the constitution $\begin{matrix} N\cdot NPh- \\ \| \\ CPh\cdot CH_2 \end{matrix}\!\!>\!C\!<\!\!\begin{matrix} -NPh\cdot N \\ \| \\ CH_2\cdot CMe \end{matrix}$ (see Feist, *loc. cit.*). With ammonia, the triketone condenses to form *2-phenyl-6-methyl-4-pyridone*, $CO\!<\!\begin{matrix} CH:CPh \\ CH:CMe \end{matrix}\!>\!NH$ (m. p. 177—178°), This compound is characterised by the fact that its aqueous solutions readily gelatinise. Several salts of the pyridone have been prepared.

157. "*The Fluorescence of Platinocyanides.*" By LEONARD ANGELO LEVY.

Barium platinocyanide exists in two forms, which exhibit a remarkable difference in their physical properties. One variety is golden-yellow and only very slightly fluorescent, the other variety being bright green and very fluorescent. The two forms are identical in crystalline character. The yellow salt is deposited from slightly acidified solutions, whereas the green form is best obtained by crystallisation from solutions containing a trace of barium hydroxide or barium cyanide. The experiments performed lead to the conclusion that the two forms have the same chemical composition, and are isomeric modifications. Similar phenomena are exhibited by the calcium salt, and to a certain extent by the cerium salt.

Barium platinocyanide, prepared from radio-active barium chloride, exhibits a strong luminosity, which is superior to that shown by a mechanical mixture of radio-active barium chloride and barium platinocyanide.

The property of fluorescence of a platinocyanide under the stimulus of radium radiations is usually exhibited in a marked degree by one hydrate form in particular. The others usually exhibit a feeble fluorescence or else show none whatever.

Certain regularities may be observed in the spectra of the fluorescence exhibited by platinocyanides of metals of the same group.

158. "*The Preparation of Disulphides.* Part II. *The Action of Alkalis on Sodium Alkyl Thiosulphates.*" By THOMAS SLATER PRICE and DOUGLAS FRANK TWISS.

The interaction between alkalis and sodium ethyl and benzyl thiosulphates in aqueous solution has been studied. The chief organic product of the action in each case is the corresponding disulphide, the main step of the reaction being probably represented by the equation $R\cdot S_2O_3Na + NaOH = RS- + Na_2SO_3 + -OH$.

The RS— residues couple, in part, to give disulphide, whilst, at all events in the case of the benzyl compound, part of the RS— residues are oxidised, sodium benzyl thiosulphate giving appreciable quantities of benzoic and thiobenzoic acids.

The results were discussed in comparison with those of Gutmann (*Ber.*, 1908, xli., 1650), who, using alcoholic solutions, has arrived at a different view of the action.

159. "*Note on the Formation of Lead Ethoxide.*" By FREDERICK MOLLWO PERKIN.

When thin sheets of lead are boiled in alcohol or suspended in the vapour of boiling alcohol, no action takes place, but if they are suspended over absolute alcohol, and ozone is bubbled through it, in a short time the surface of the lead becomes tarnished, and then assumes a brownish yellow colour. At the end of some days, the whole of the lead is converted into a light yellow powder. On carefully washing this powder with alcohol and placing in an evacuated desiccator over sulphuric acid for some hours, a light yellow, friable powder is obtained. This, on analysis, proves to be *lead ethoxide*, $Pb(OEt)_2$. It appears to be soluble in water, although a clear solution is never obtained, probably owing to hydrolysis. It has not been found possible to obtain the ethoxide by passing oxygen alone through the alcohol, but, after the ozone has been passed for some time, the reaction can be completed by the continued passage of dry oxygen. When mercury is placed in alcohol and ozone bubbled through it, a white powder is formed which has not yet been analysed. The method is being tried with other metals.

160. "*Some Reactions of Phenylhydrazine with Metallic Cyanides and other Salts.*" By ROBERT DE JERSEY FLEMING STRUTHERS.

Phenylhydrazine in alcoholic solution combines with cuprous cyanide in ammoniacal solution to form an insoluble compound, $2CuCN,3C_6H_5\cdot NH\cdot NH_2$. This, superficially, closely resembles the compound obtained (*Proc.*, 1905, xxi., 95) by heating cuprous cyanide in an excess of phenylhydrazine, being similarly composed of shining scales, which gradually oxidise in air, and acquire a brilliant coppery sheen. These scales consist of microscopic crystals, the angles of which have now been successfully measured.

The substance readily decomposes on heating, nearly according to the equation :—

$$2(2CuCN,3C_6H_5\cdot NH\cdot NH_2) = 4CuCN+3N_2+3C_6H_6+3C_6H_5\cdot NH_2+3NH_3.$$

This, together with the previously described compound, $CuCN,C_6H_5\cdot NH\cdot NH_2$, co-ordinates with Moitessier's series of three compounds of phenylhydrazine with cuprous chloride, bromide, and iodide respectively (*Bull. Soc. Chim.*, 1899, [3], xxi., 666), and fills up the gaps, as below :—

$2Cu_2(CN)_2,4C_6H_5\cdot N_2H_3$; $2Cu_2Cl_2,5C_6H_5\cdot N_2H_3$,
$2Cu_2(CN)_2,6C_6H_5\cdot N_2H_3$; $2Cu_2Br_2,7C_6H_5\cdot N_2H_3$.
$2Cu_2I_2,8C_6H_5\cdot N_2H_3$.

Cobalt cyanide is found to exert a powerful catalytic action on phenylhydrazine, 0·03 to 0·04 grm. sufficing to determine the decomposition of 5 or 6 cc. of phenylhydrazine with almost explosive violence. The mixture is heated near its boiling-point for a short time until action is thoroughly established. The flame is then removed, but the action progresses with increasing intensity, and much evolution of heat, until the phenylhydrazine is completely decomposed. This catalysis by cobalt may be kept under control if only a few mgrms. of the cyanide are added to 4 or 5 grms. of phenylhydrazine, and the mixture distilled over paraffin, keeping the temperature carefully as low as 200°. In this way the evolved nitrogen may be easily collected and measured. The products appear to be principally aniline, benzene, ammonia, and nitrogen.

Nickel cyanide has a similar, but less, powerful action. Other salts of cobalt and nickel appear also to act more or less as catalysts. It seems possible that reduction may first occur, and the reduced metal be the true catalyst, although metallic nickel, not very finely divided, had only a slight effect. This action of cobalt is much more powerful than that of cuprous cyanide as described in the previous paper.

Other salts of copper have a strong catalytic action; cupric chloride is nearly comparable with cobalt cyanide, and finely divided metallic copper (precipitated by iron) is fairly powerful as a catalyst. The acetate and sulphate, which deposit the metal as a mirror, are less powerful. Other metallic salts, such as lead and bismuth, seem to exert less action. In the case of lead acetate, a mirror of lustrous and coherent lead was deposited on the glass, resembling the copper mirror obtained by Chattaway, as described by him recently.

161. "*The Formation of Polyiodides in Nitrobenzene Solution.* Part III. *The Chemical Dissociation of the Polyiodides of the Alkali Metals and Ammonium Radicles.*" By HARRY MEDFORDTH DAWSON.

A method has been devised by means of which the chemical dissociation in nitrobenzene solutions containing iodine and iodides of the alkali metals and substituted ammonium bases can be measured. This consists in the determination of the composition of the aqueous solutions which are in equilibrium with nitrobenzene solutions containing known quantities of iodide and iodine; from relationships which have been previously established for aqueous iodide-iodine solutions, the concentration of the free iodine in the aqueous solutions can be calculated, and from this the concentration of the free iodine in the nitrobenzene solutions is obtained.

It was shown that polyiodides of the types MI_9, MI_7, MI_5, and MI_3 exist in nitrobenzene solutions, and that these represent the successive steps in a dissociation phenomenon in which iodine is the other product of dissociation. In solutions of the same molar concentration, the polyiodides of the different alkali metals and ammonium bases are chemically dissociated to the same extent; this is probably due to the fact that the polyiodides are largely ionised, and the iodine resulting from the dissociation of a complex iodine ion which is common to the various polyiodides. Whereas the higher polyiodides are chemically dissociated to a considerable degree, the tri-iodides are very stable, and are but very slightly dissociated.

162. "*The Hydrolysis of Amygdalin by Emulsin.*" (Part II.). By SAMUEL JAMES MANSON AULD.

By observation of the change of rotation of the dextrose produced during the hydrolysis of mandelonitrile glucoside by emulsin, and the approximate constancy of the rotation after hydrolysing amygdalin by emulsin, it is possible to show that amygdalin is derived from an $\alpha\beta$-disaccharide, the β-dextrose residue being attached to the benzaldehyde-cyanohydrin nucleus.

Exact measurement of the quantities of hydrocyanic acid, benzaldehyde, and dextrose formed from amygdalin shows that mandelonitrile glucoside is formed as an intermediate product during the hydrolysis by emulsin, the biose ether-linking breaking preferentially. By stopping the hydrolysis at a suitable point, it is possible actually to isolate Fischer's glucoside.

163. "*A New Form of Potash Bulb.*" By ARTHUR EDWIN HILL. (Will be inserted in next issue).

CORRESPONDENCE.

THE RECENT LETTERS OF THE HONORARY SECRETARIES OF THE CHEMICAL SOCIETY.

To the Editor of the Chemical News.

SIR,—The Fellows of the Chemical Society have recently received two circular-letters signed by the Honorary Secretaries. The motive of the letters has been, primarily, the reception by the Council of a petition for the admission of women to the Fellowship. It is not with the object of supporting that petition, which I have signed, that I write this letter, but in the hope of helping to stop the further issue of letters such as these over the official signatures of the Society's Honorary Secretaries.

The first of the two letters was authorised by the Council; but it may be confidently assumed that had the Council foreseen how that letter would be composed, it would not have adopted it. It must by this time be evident, even to the members present at the last Council Meeting, that, as carried out, the resolution to issue to the Fellows, not a copy of the petition, but a statement instead of arguments for and against the admission of women, has proved itself to be unwise. We are told that the petition was not sent out, for fear that it might bias the opinion of Fellows—as though the Council had jurisdiction over the opinions of the Fellows, and with it the duty of guarding these opinions from being made other than a reflex of its own.

Half the Members of Council had signed the petition, yet those who had the ear of the meeting persuaded it to withhold the petition from the Fellows, on the ground that it was calculated to gain their adhesion to the movement—as, indeed, and quite properly, it was. I will not believe that, in saying that the petition would "bias" the opinion of Fellows, the Honorary Officers, in the second letter, meant to impugn the honorable and open character of a petition signed and supported by numbers of eminent chemists. Thus, inadvertently without doubt, the Council meeting passed a resolution calculated to affect unfavourably the votes of the Fellows, by withholding the petition from them. Before, therefore, the request to the Fellows for their votes had gone out, the opponents of the measure

had gained an unfair advantage, and they managed to gain still more.

The adoption of the alternative, that of sending out a statement of arguments on both sides, made the Council responsible for a still more seriously objectionable action by its Secretaries, for, as already pointed out, the Council can claim no right to bias or influence the opinions of the Fellows, or to submit to them a partial statement concerning a petition it has seen fit to act upon. The Council is responsible—but hardly perhaps to blame—for what was done; yet surely the meeting might have recognised the moral impossibility of an impartial statement being drawn up by men so strongly biassed and so active in opposition as the Honorary Secretaries were known to be. The consequence of this mistaken trust in the capacity of its Secretaries to carry out a delicate commission is already well known. The Secretaries omitted half the preamble of the petition. They said that the petition was for an inquiry, but they left out that it rested on the opinion that the time had come when the Fellowship of the Society should be rendered accessible to women. That is the published opinion of half the members of the Society's Council and of very many chemists of repute besides, yet the Fellows were to be left in the dark as to that. The serious consequence of this mangling of the preamble is emphasised by the circumstance that the statement had already been circulated, that many who had signed the petition had done so only for an inquiry, and not from a conviction that women should be eligible for the Fellowship. It cannot be pleaded that the Secretaries had not in mind the insertion of the preamble or any part of the petition in their letter, for in the second letter they give the latter half of the preamble within inverted commas, still leaving out its first half.

For issuing to the Fellows a statement of the arguments for admitting women to the Society, the Council Meeting had the petition in its hands to offer in justification of its action. But what had it to justify its sending out arguments against admitting women, not advanced by itself; but by "those who are unwilling" to admit women? From whom had the Council Meeting obtained official cognisance of the nature of these arguments? It had received no counter petition, and it did not itself advance these arguments. Did the Council, when acceding to the request of the petition to appeal to the Fellows, act constitutionally in publishing against the object of the petition arguments which it had not itself official knowledge of and had not endorsed? In any case, did it not, by issuing these statements of unindicated and unknown persons, act prejudicially towards the object of the petition, without finding it to be its duty to directly oppose that object. Still, again, advantage was given to the opposition by the Meeting's resolution to submit arguments for and against, instead of the petition itself.

The second letter, the receipt of which has led me to write this protest, cannot be denounced in the terms it deserves, since it is signed by all the Honorary Officers, including the President, whose impartiality is above suspicion. As the Fellows know, what led up to the publication of that letter, dated July 8th, was the letter to the Fellows from eleven Members of Council, Past Presidents of the Chemical Society. The Fellows, whatever view they may each hold on the subject of the Fellowship, must certainly feel greatly indebted to these Past Presidents, as also to Sir Henry Roscoe for his letter to *The Times*, July 3rd, to Professors Dixon and Perkin for theirs to the *Manchester Guardian*, July 4th, and to the writer of the article in *Nature* of July 9th, for putting the petition or the substance of it before them. The Past Presidents, it is to be noted, date their letter from Oxford, sign it without titles, word it most modestly, and make no reflection in it upon the action of the Council.

The Honorary Officers have thought it right to write to the Fellows from the Offices of the Society, with official signatures, and with the formality of an authorised communication from the Council, a letter charging the Past Presidents with having adopted a course unauthorised by the Council, and in direct opposition to a resolution passed by it, and to have done this in a way which might well have led Fellows to conclude that the Council had authorised their procedure. It is difficult to realise that such an apparently insulting and, at the same time, absurd charge against eleven most eminent and honoured chemists and Members of the Council has been made by the Officers, and, of course, it could not have been meant to be what, literally taken, it is. The Officers, we know, had no authority to write the letter as Officers of the Society; on the other hand, the Past Presidents' letter is not, on any possible view of it, open to the charge of assuming or claiming any authority but their own. What must have been the frame of mind of the Secretaries when writing a letter to the Fellows not only implying that the Past Presidents, in publishing the petition, required the authority of the Council or its Officers for this most serviceable and important action, but also representing that the Past Presidents had issued the petition in a way liable to lead to the belief that their action had the authority of the Council, regardless of the facts that the covering letter was dated from Oxford, and that the signatures without titles were those of universally known and respected and honourable gentlemen.

Everyone recognises the chemical ability and the estimable qualities of the Honorary Secretaries, but there are some who are now expressing doubt whether they should be fully entrusted with the writing of the Council's letters.—Iam, &c.

EDWARD DIVERS.

London, July 14, 1908.

PROPOSED ADMISSION OF WOMEN TO THE FELLOWSHIP OF THE CHEMICAL SOCIETY.

THE following letter has been sent to all Fellows of the Chemical Society:—

Chemical Society,
Burlington House, London, W.
July 8th, 1908.

DEAR SIR,—You have doubtless received a letter emanating from Oxford and dated July 1st, enclosing a circular copy of the petition recently presented to the Council of the Chemical Society requesting the Council "to take such steps as may appear desirable to ascertain the wishes of the Society as a whole in regard to this question."

The envelopes enclosing the above communications bore the familiar printed address-slips which might well lead Fellows to conclude that they were issued by authority of the Council. This is not the case.

The Council resolved that the statement of arguments on both sides which was issued to Fellows on June 23rd, was better calculated to elicit the unbiassed opinion of all Fellows if sent alone.

The course adopted by the signatories of the Oxford Circular of July 1st is therefore not only unauthorised, but is in direct opposition to a resolution of the Council of which the signatories are members.—We are, Dear Sir, Yours faithfully,

(Signed) WILLIAM RAMSAY, President
HORACE T. BROWN, Foreign Secretary.
ALEXANDER SCOTT, Treasurer.
M. O. FORSTER, ARTHUR W. CROSSLEY } Secretaries.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 6th inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, in the Chair. Lady Low and Mr. H. N. Middleton were elected Members. The Special Thanks of the Members were returned to Mr. Oswald Lewis for his donation of £5 5s. to the Fund for the Promotion of Experimental Research at Low Temperatures.

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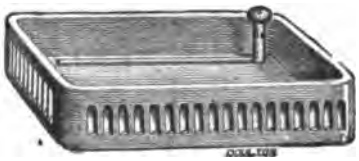

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THE

CHEMICAL NEWS

AND

JOURNAL OF PHYSICAL SCIENCE

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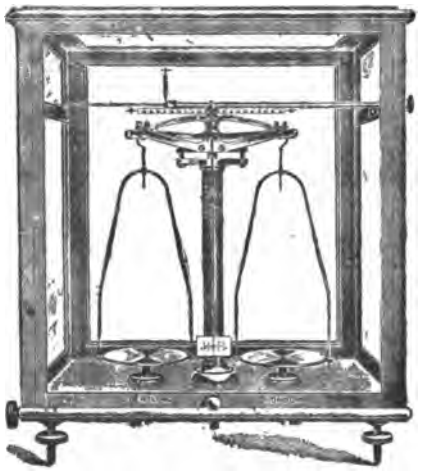

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THE CHEMICAL NEWS.

Vol. XCVIII., No. 2539.

THE LIQUEFACTION OF HELIUM.

We are now in a position to give the corrected version of the achievement of Professor Onnes, which carries with it the explanation of his wish for delay in publication. On July 14 he announced the liquefaction of helium in the following telegram, which was placed at our disposal by the courtesy of Sir James Dewar, who replied with "hearty congratulations on splendid achievement":—

Leiden, July 14.

Dewar, Royal Institution, London.

Obtained 10 July 60 cubic centimetres liquid helium. Lasted for two hours. Not solid at two millimetres vapour pressure.

Kamerlingh Onnes.

On July 15 Professor Onnes telegraphed as follows, and also *ex abundante cautela*, telegraphed directly to ourselves, as already stated, a request for delay of publication:—

Leiden, July 15.

Dewar, Royal Institution, London.

Hearty thanks. Will give you great satisfaction to see considerable part joint to Van der Waal's theory your methods take in success, but please wait publication *Times* till last controls are ready.

Kamerlingh Onnes.

On the 18th, having verified his results, and probably repeated his experiment, he gave his final version in the following telegram:—

Leiden, July 18.

Dewar, Royal Institution, London.

Please change two millimetres into one centimetre; can then be published in *Times*. Boiling-point 4·3.

Kamerlingh Onnes.

Professor Onnes has therefore shown that helium is a liquid having a boiling-point of 4·3° absolute, which is not solid when exhausted to a pressure of 10 millimetres of mercury, at which pressure the temperature must have been reduced to within 3° of the absolute zero—*i.e.*, about one-fourth of the temperature of hydrogen in corresponding conditions, as that again is about one-fourth of the corresponding nitrogen temperature. If we could obtain another similar drop by the discovery of a gas still more volatile than helium we should have a liquid boiling about 1° above the absolute zero.

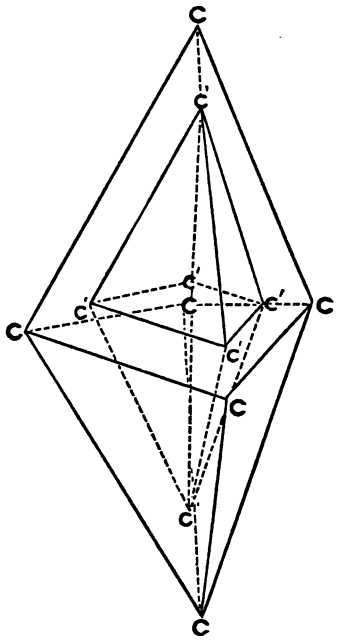
A few notes upon the steps by which the liquefaction of helium has been reached may be of interest. In 1895, by the application of the method of sudden expansion from high compression, Olscevski, starting from the temperature of exhausted air, failed to get any appearance of liquefaction. In 1901, Dewar, in the Bakerian Lecture, described his repetition of that experiment, using liquid hydrogen under exhaustion instead of liquid air, again without obtaining any trace of condensation. Reasoning from the analogy of his experiments on the liquefaction of hydrogen, he showed that by regenerative cooling starting from the temperature of liquid hydrogen, we might expect to liquefy a gas whose boiling-point might be as low as 4° or 5° absolute. In his Presidential Address to the British Association in the following year he gave reasons for placing the boiling-point of helium at that figure, showing at the same time how great are the experimental difficulties of getting within 5° of absolute zero. In 1905, Olscevski repeated Dewar's experiment of 1901, using higher pressures, and reached the conclusion that the boiling-point of helium must be below 2° absolute, and that after all the gas might be permanent. The same experiment was repeated early in 1908 by Professor Onnes with a much larger quantity of helium than had previously been available, and at first thought he had obtained solid helium, but found that the appearance was due to impurity in the gas. Dewar again repeated the experiment by circulating helium in a regenerative apparatus, but though he got cooling, he was baffled by the inadequacy of his supply of helium to maintain the cooling process sufficiently long to reach liquefaction. At last, by the experiment of July 10, Professor Onnes has definitely settled the matter. As new and richer sources of helium have been discovered, and its separation has been enormously facilitated by Dewar's charcoal method, it is possible that helium may become sufficiently abundant in cryological laboratories to be used as liquid hydrogen is now used in physical research.—*The Times*, July 20.

THE CONSTITUTION OF THE CARBON MOLECULE.

By J. C. THOMLINSON, B.Sc.

In the Chemical News of Jan. 24th (vol. xcvii., p. 37) I noted a critique of Sir James Dewar's suggested formula for the constitution of the carbon molecule.

I do not go into the question of the reasoning which in a former paper, Jan. 6th, 1906 (Chemical News, xcii., p. 37), and succeeding issues, lead me to give suggested heats of combination for carbon and other elements as distinct from the heat they evolve on union with oxygen or the heat of combustion, I would merely call attention to the accompanying diagram as representing a twelve



atom molecule of carbon as it might actually occur in space.

Without actually giving the geometric form of the system to which the diamond belongs, it will be seen that two solid figures, one enclosed within the other, and each containing six solid angles, could easily represent the position of twelve atoms of carbon which form the molecule.

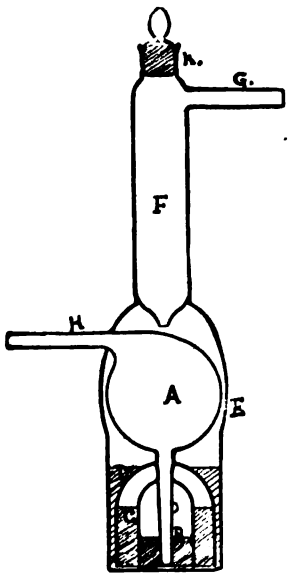
Such a figure comes within the terms of the description of the formation of the carbon molecule from two benzene nuclei as described in the above-mentioned critique, for we can easily imagine either of the solid figures given as formed from a benzene molecule by the elimination of the hydrogen atoms with the strengthening of the bonds of valency of the carbon atoms among themselves, these valencies being represented by the sides of each solid figure respectively.

NOTE.—Intramolecular tension mentioned in the article already quoted in connection with the discrepancies in the results of theory and practice would in the spacial formula given be accounted for by any force (physico-chemical) acting between the carbon atoms other than that represented by the usual valency bonds.

A NEW FORM OF POTASH BULB.*

By ARTHUR EDWIN HILL.

THE potash bulb indicated in section in the figure consists essentially of the inlet tube A B, made up of the bulb A, and its lower extension the narrow tube B, which extension is surrounded by, and fused to, the glass domes C and D. Enclosing these is the outer vessel E, to which H, the upper part of the inlet tube, is fused. The exit tube F, which is drawn off at its lower end to serve as a receptacle for holding soda-lime, is fused to the top of the vessel E, and provided with a ground stopper K, and side outlet tube G.



To prepare the potash bulb for use, the ground stopper K is withdrawn, and a small plug of glass-wool is inserted and pushed to the bottom of the tube F. Recently ignited soda-lime, free from fine particles, is then poured into the tube until it is three parts full, and the remaining space is filled with small granules of calcium chloride; a small plug of glass-wool is inserted at the top, and the ground stopper is replaced. One such filling will last for at least twenty experiments.

The bulk of the carbon dioxide is absorbed with a concentrated solution of potassium hydroxide, which is contained in the vessel E, and renewed after every experiment. Any residual traces of carbon dioxide are removed by the soda-lime in the tube F, which also assists the calcium chloride to desiccate the gases before they leave the potash bulb.

A special feature of the potash bulb, which makes it very efficient, is the introduction of the domes C and D, which cause the gases to bubble through the potash solution three times in succession before they can come into contact with the soda-lime in the tube F.

After the gases have entered the bulb A, they pass down the narrow tube B, and bubble up through the solution into the dome C; they leave this through two holes in its wall, and bubble up through more solution into the outer dome D, which they then leave through two holes to enter the outer vessel E, after bubbling through the solution surrounding the dome D.

The position of these holes is so arranged that the gases must bubble through at least a quarter of an inch of potash solution each time; sufficient solution must be introduced into the potash bulb to cover the top of the dome D when the gases are bubbling through the solution. The section of the dome D is made elliptical in order to increase the quantity of solution adjoining the holes through which the gases bubble.

Although this bulb was designed for absorbing carbon dioxide, it can also be used for absorbing water vapour. For this purpose, the tube F is filled with granulated calcium chloride, and concentrated sulphuric acid is substituted for potassium hydroxide solution in the vessel E.

This new form of potash bulb presents a number of advantages.

1. A treble scrubbing action, which ensures a very complete absorption by means of a comparatively small quantity of liquid absorbent, supplemented by a final scrubbing with soda-lime and calcium chloride, which also ensures complete desiccation.

2. It is very compact and strong, being free from most of the weaknesses which characterise many of the potash bulbs now in use. It is easily manipulated, and will stand on its own base.

3. It can be easily and rapidly wiped dry before it is weighed, and will therefore give thoroughly concordant results, which are not affected by the amount of moisture in the surrounding air.

(We are indebted to the author for permission to use the woodcut illustrating this Paper).

* Abstract of a Paper read before the Chemical Society, June 18, 1908.

DETERMINATION OF NICKEL AND CHROMIUM IN STEEL.

By EDWARD DEMILLE CAMPBELL and WALTER ARTHUR.

IN February, 1895, one of the authors in conjunction with W. H. Andrews, published a method for the determination of nickel in nickel steel (*Journ. Am. Chem. Soc.*, xvii., 125), in which the nickel was determined electrolytically or by titration with potassium cyanide, using silver iodide in suspension as an indicator. About six months later Thomas Moore published his article "The Volumetric Estimation of Nickel" (CHEMICAL NEWS, lxxii., 92), in which he showed that the use of silver iodide as an indicator renders the cyanide titration of nickel sharp and accurate. Credit for the cyanide titration of nickel by the use of silver iodide as an indicator is usually given to Moore, and undoubtedly this modification was worked out independently. In most of the materials analysed by Moore the proportion of iron to nickel was comparatively small. He makes the statement towards the latter part of his article that "In presence of alumina, magnesia, or ferric oxide, citric acid, tartaric acid, or pyrophosphate of sodium may be employed to keep them in solution."

A number of volumetric methods based upon the cyanide titration of nickel using silver iodide as an indicator have appeared since the above was published. Most of these methods aim either to remove the iron before titrating for nickel or to add some reagent which will keep the iron dissolved when the solution is rendered alkaline.

H. Brearley (CHEMICAL NEWS, lxxiv., 16) precipitates the iron in rather strongly acid acetate solution as basic acetate, and titrates the nickel in an aliquot part of the filtrate, while G. T. Dougherty (*Iron Age*, April 25, 1907) precipitates iron as ferric hydroxide by means of a large excess of ammonia in presence of a strong solution of ammonium chloride. His results seem to show that if sufficient ammonium chloride be added, the ferric hydroxide will not contain any nickel, so that the titration of the aliquot portion of the filtrate will give accurate results.

As early as 1899, G. W. Sargent (*Journ. Am. Chem. Soc.*, xxi., 854) described a combination of the now well-known ether method for the separation of iron with the cyanide-iodide titration for nickel. This combined method has been quite satisfactory in many ways, but where a large number of determinations are being made, the expense of the ether, combined with the danger of fire attending its use and the disagreeable effect of inhaling the vapours, render the use of the ether method not entirely satisfactory.

The latest modification of the cyanide-iodide method for nickel in steel is that proposed by C. M. Johnson (*Journ. Am. Chem. Soc.*, xxix., 1201). Johnson describes the cyanide-iodide method as that of T. Moore, although this method appeared in this country some six months in advance of the latter's article.

In the method proposed by Johnson, which is a modification of that given by Brearley and Ibbotson in their "Analysis of Steel Works Materials," p. 183, the iron is kept in solution in the presence of a little free ammonia, by means of a large amount of citric acid, instead of a small amount as recommended by the above authors. By using 12 grms. of citric acid per grm. for ordinary nickel steels and 24 grms. in case of chrome-nickel steels, the colour of the solution is rendered light enough to observe the silver iodide without difficulty.

Johnson has shown that the amounts of vanadium, tungsten, molybdenum, and manganese found in commercial steels do not interfere with the titration of nickel, and that the dark colour due to the presence of chromium may be partially overcome by the use of an increased amount of citric acid.

Experiments made in the laboratory of the University of Michigan with the method described by Johnson showed that even in the presence of large amounts of ammonium citrate, the brownish colour of the ammoniacal solution did not permit as satisfactory a titration of nickel to be made as desired. We have endeavoured therefore to see whether the use of sodium pyrophosphate to keep iron in solution would not give better results than the ammonium citrate.

In his original publications, Moore gave no details concerning the use of sodium pyrophosphate, since in the materials on which he worked the amount of iron present was not large enough to cause any trouble. In applying the method to the analysis of steel, some care is required to obtain conditions, such that the colour of the solution will not be dark enough to cause any uncertainty as to the end reaction. The following method has been worked out, and found to give satisfactory results in this laboratory:—

One grm. of the steel is dissolved in a casserole with 10 to 15 cc. of nitric acid (sp. gr. 1·2) with the addition of a little hydrochloric acid if necessary. After solution of the steel, 6 or 8 cc. of sulphuric acid (1:1) are added, and the solution is evaporated until the sulphuric anhydride fumes begin to come off. Thirty to 40 cc. of water are now added, and the solution heated nearly to boiling or until the ferric sulphate is all dissolved. The solution is then transferred to a 400 cc. beaker. In case the solution in the casserole is not perfectly clear but contains a little silica or carbon in suspension, it should be filtered so that the solution in the beaker is perfectly clear and bright. To the sulphuric acid solution in the beaker, which should be about 60 or 70 cc. in volume, are added 13 grms. of sodium pyrophosphate previously dissolved in about 60 or 70 cc. of water heated to about 60° or 70°. After the addition of the sodium pyrophosphate, the solution containing the heavy white precipitate of ferric pyrophosphate is cooled to room temperature, and dilute ammonia (1:1) is added drop by drop, while the solution is vigorously stirred, until the greater portion of the pyrophosphate has dissolved, and the solution has assumed a greenish tinge. The solution at this point should have an alkaline reaction towards litmus, but it should not smell of free ammonia. The solution is now warmed gently, and stirred, when the remaining pyrophosphate will competely dissolve, giving a perfectly clear light green solution. If the ammonia is added too fast or the solution is not carefully stirred, a brownish colour is apt to result, but this can usually be overcome by carefully adding a few drops of dilute sulphuric acid. The clear light green solution is now cooled to room temperature, and 5 cc. of silver nitrate (made by dissolving 0·5 grm. of silver nitrate in 1000 cc. of water) are added, followed by 5 cc. of a 2 per cent solution of potassium iodide. Standard potassium cyanide is now run in until the clearing up of the solution shows that the nickel has been completely converted into potassium nickel cyanide and the silver iodide into potassium silver cyanide. Before calculating the amount of nickel present, correction must be made for the amount of potassium cyanide required for the 5 cc. of indicator used.

The potassium cyanide used may be decinormal, made by dissolving about 13 grms. of potassium cyanide in 1000 cc. of water, and standardising by direct titration with standard decinormal silver nitrate. If decinormal potassium cyanide is used, since the titration of the nickel takes place according to the equation $NiSO_4 + 4KCN = K_2Ni(CN)_4 + K_2SO_4$, the decinormal factor of metallic nickel will be 0·002935. If preferred, the potassium cyanide may be standardised against a nickel solution of known strength.

The above method gives very satisfactory results with nickel steels not containing chromium or copper.

If copper is present in amounts not exceeding 0·4 per cent, the amount of potassium cyanide required for the copper will not be as great as it would for an equal per cent of nickel, but the copper will replace almost exactly three-quarters of its weight of nickel. Thus, if a sample contained 0·12 per cent of copper, it would require an amount of potassium cyanide equivalent to 0·09 per cent of nickel. In almost all cases of nickel steels containing copper, if the sample is dissolved in a covered Erlenmeyer flask in 25 cc. of water to which is added 5 cc. of sulphuric acid (sp. gr. 1·84), the nickel will all go into solution, and the copper will remain undissolved. The solution is cooled, and the undissolved copper is filtered out. The ferrous sulphate in the filtrate is oxidised by the addition of a slight excess of hydrogen peroxide, and the excess of this latter is boiled off. The solution is concentrated if necessary, and the method continued as in the case of nickel steel containing no copper.

In the case of chrome-nickel steels the sample is dissolved and converted into sulphate as described in the first method. After dissolving in the casserole in about 40 to 50 cc. of water, the solution is brought to boiling and a 2 or 3 per cent solution of potassium permanganate is added drop by drop to the boiling solution until a slight permanent precipitate of manganese dioxide is obtained. The chromium in this way is readily oxidised to chromic acid much as in the well-known Galbraith process for the determination of chromium. The solution is now cooled to room temperature, filtered, and the filtrate and washings concentrated in a 400 cc. beaker to about 60 cc. Sodium pyrophosphate is added, and the process continued as in the first instance when chromium is not present. By oxidation of chromium to chromic acid, the dark colour due to the presence of chromic salts is avoided, and the chromic acid takes no part in the titration of the nickel. The clear solution to which silver nitrate is added, will have a yellow rather than a green colour when chromium is present, but no trouble is experienced in obtaining a sharp end reaction.

In case of chrome-nickel steels, the chromium may be advantageously determined in the same sample as that used for the determination of the nickel. The solution of the

sulphates in which the chromium has been oxidised to chromic acid, must be cooled thoroughly before filtering if filter-paper is used, in order to avoid any reduction of the chromic acid by the paper. To the clear solution of the sulphates containing chromic acid, an accurately weighed amount, usually 0·7 grm., of ferrous ammonium sulphate is added, and the excess of ferrous sulphate oxidised by means of standard potassium permanganate.

In order to obtain an accurate titration by means of potassium permanganate in the presence of chromic sulphate, it is best to weigh out the ferrous ammonium sulphate and reserve a small portion to be added after the pink colour of the permanganic acid, usually indicating the end reaction, has been obtained. The reserved portion of the ferrous ammonium sulphate is then added, and the titration continued, employing, however, potassium ferricyanide as an external indicator as in the titration of iron by means of potassium dichromate. Since the green chromic sulphate is partially complementary to the red permanganic acid, the colour of this latter which usually indicates the end reaction will not be visible until too large an excess has been run in. From the weight of the ferrous ammonium sulphate used and the amount of the decinormal potassium permanganate required, the per cent of chromium may be readily calculated as in the well-known methods for the volumetric determination of this element. When chromium has been determined, the solution is again brought to the boiling-point, and the chromium again oxidised to chromic acid by the addition of a 2 per cent solution of potassium permanganate until a slight permanent precipitate of manganese dioxide is obtained. The solution is again cooled until cold, and the manganese dioxide filtered out, and the method continued as though the chromium had not been determined. If more than 1 grm. of ferrous ammonium sulphate has been used, 1 or 2 grms. additional sodium pyrophosphate should be added.—*Journal of the American Chemical Society*, xxx., No. 7.

THE ACTION OF HYDROGEN SULPHIDE ON ALKALINE SOLUTIONS OF ZINC SALTS.

By L. W. McCAY.

THE fact that the zinc sulphide, or the zinc hydrosulphide, precipitated from alkaline solutions of zinc salts by sodium or potassium hydrosulphide is soluble in an excess of these reagents, and that the zinc sulphide, or zinc hydrosulphide, precipitated from alkaline solutions of the metal by hydrogen sulphide dissolves when the gas is permitted to act on the solutions for some time, appears to have escaped the notice of the analytical chemists.

NOTE.—I noticed this peculiar behaviour of zinc sulphide some three or four years ago, and supposed that my observation was a new one. I found, however, that the reaction was first observed by Julius Thomsen in 1878 (*Ber.*, xi., 2044) and subsequently examined by A. Villiers (*Comptes Rendus*, cxx., 97). Lottermoser ("Samlung chem. u. tech. Vorträge," VI.) and Winsinger (*Bull. de l'Acad. des Sciences de Bruxelles*, [2], xiv., 321) also refer to it.

At all events, in no work on analytical chemistry to which I have access is this remarkable behaviour of zinc sulphide referred to. The solution of the zinc sulphide is a colloidal one, for the zinc in it will not pass through parchment paper. The zinc sulphide, or zinc hydrosulphide, acts towards sodium and potassium hydrosulphides in much the same way that zinc oxide, or zinc hydroxide, acts towards sodium and potassium hydroxides. The analogy between the two reactions almost compels one to conclude that the change takes place in the sense of the equation $Zn(SH)_2 + 2RSH = R_2ZnS_2 + 2H_2S$.

If, however, an alkali sulphozincate is actually formed, t must be very unstable, for concentrated solutions of mineral salts, when added to its solution, precipitate only zinc sulphide, or possibly zinc hydrosulphide; and then too the zinc sulphide separates out gradually, and in a slimy condition, when the solution is allowed to stand.

In many books on qualitative analysis the student is directed to separate manganese from zinc by adding to the solution of their chlorides an excess of sodium or potassium hydroxide. Now if the alkaline solution of the zinc, after its separation from the manganous hydroxide, be treated for fifteen to twenty minutes with a rapid current of hydrogen sulphide, the zinc sulphide which is first precipitated may dissolve. The smaller the amounts of zinc and sodium chloride present in the solution, and the more rapid the current of gas, the more readily does the zinc sulphide formed pass into solution. Should a student pass a rapid current of hydrogen sulphide into such a solution, and then leave the spot and not return until after the lapse of some fifteen to twenty minutes, the chances are he will find the solution clear, or nearly so, and report no zinc. I have convinced myself by a number of experiments that there is, in a case of this sort, considerable danger of overlooking the zinc. It is a significant fact that Fresenius uses hydrogen sulphide water in order to test for the zinc in the alkaline solution ("Anleitung z. Qualit. Chem. Analyse," 1874, 291). He is also careful to state that an excess of the reagent is to be avoided. However, in describing the special reactions of zinc he states that hydrogen sulphide precipitates from alkaline solutions all the zinc in the form of the hydrated sulphide (*loc. cit.*, p. 137). Nothing is said about an excess of the reagent, although he does mention the fact that ammonium chloride greatly promotes the separation of the precipitate.

In the following experiments I used a sodium hydroxide solution of zinc oxide containing 8 grms. of the oxide in a litre:—

1. Ten cc. of the solution were diluted to 150 cc. and treated with a rapid current of hydrogen sulphide. The zinc sulphide was precipitated almost immediately, but at the end of fifteen minutes it had passed into solution.

2. To 10 cc. of the solution, made faintly acid with hydrochloric acid, a few drops of a concentrated solution of manganous sulphate were added and the metals precipitated as sulphides with yellow ammonium sulphide. After filtering, dissolving the precipitate in a small amount of very dilute hydrochloric acid, and separating the manganese with excess of sodium hydroxide, the alkaline filtrate was diluted to about 150 cc. and treated with a rapid current of hydrogen sulphide. In fifteen minutes the zinc sulphide had dissolved and the solution was clear.

3. The experiment was repeated with a solution containing the same amount of zinc oxide along with considerable amounts of manganese, cobalt, and nickel. The alkaline solution of the zinc oxide, however, behaved toward the sulphuretted hydrogen gas exactly as it did in 1 and 2.

On standing, all three solutions became turbid, owing to a gradual separation of the zinc sulphide.—*Journal of the American Chemical Society*, xxx., No. 3.

Preparation of Argon from the Air by means of Calcium Carbide.—Franz Fischer and Oskar Ringe.—In this paper the authors describe in detail their new method of preparing argon from the air, which gives good yields of the gas at a comparatively low cost. The oxygen and nitrogen are absorbed by means of a finely powdered mixture of 90 per cent calcium carbide and 10 per cent calcium chloride, the absorption being carried out at 800° in an iron vessel specially constructed for the purpose. An apparatus for insuring thorough circulation of the air has been devised, and by means of it 11 litres of argon can be obtained by the aid of 7 kilogrms. of carbide in two days. The density of the gas was determined and found to be 19·94 (O = 16).—*Berechte*, xli., No. 10.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 33).

Tabular Outline.

IN the accompanying tables the enclosure of a symbol in brackets shows that the element may divide itself between the residue and the solution in the operation immediately preceding.

Procedure and Notes.

Procedure 51.—Boil the filtrate from the H_2S precipitate (P. 21) till the H_2S is expelled. (For this and similar references to former procedures, see the previous article, *Journ. Am. Chem. Soc.*, xxix., 137). Transfer it to a flask, add NH_4OH (0·96) until the mixture after shaking smells of it, and then 2 to 4 cc. more. Note whether there is a precipitate. Add ammonium monosulphide slowly (or if nickel is likely to be present pass in H_2S), until, after shaking, the vapours in the flask blacken a piece of filter-paper moistened with lead acetate solution. To coagulate the precipitate shake the mixture, or heat it nearly to boiling. Filter, and wash the precipitate, first with water containing about 1 per cent of the $(NH_4)_2S$ reagent, and then with a little pure water. If the filtration is slow, keep the funnel covered with a watch-glass so as to prevent oxidation. To the filtrate add a few drops $(NH_4)_2S$, boil the mixture for a few seconds, or longer if it is dark coloured (till it becomes colourless or light yellow); filter if there is a precipitate, uniting it with the preceding one. (Precipitate, P. 52; filtrate, P. 71, or first by *P. 51*a*, if vanadium is to be tested for).

Notes.

1. The H_2S is boiled out, and the effect of the addition of NH_4OH alone is noted because it often gives a useful indication as to what elements are present. To save time the expulsion of the H_2S may be omitted when this indication is considered unimportant. Ammonium monosulphide is used, rather than polysulphide, in order to prevent as far as possible the dissolving of NiS, and in order not to introduce sulphur into the precipitate, or polysulphide into the filtrate; for this gives to the filtrate a deep yellow colour, and causes in the subsequent analysis separation of sulphur on standing or on heating. Excess of the monosulphide is avoided for the same reasons, since it rapidly oxidises in the air to polysulphide. By passing H_2S into the ammoniacal solution instead of adding $(NH_4)_2S$, the dissolving of NiS is entirely prevented; therefore, though the operation takes a little longer, the use of H_2S is to be preferred when nickel is likely to be present. The mixture is shaken in order to coagulate the precipitate and make it filter more readily. Heating also promotes the coagulation of the precipitate, and it is recommended when the precipitate does not coagulate and settle quickly on shaking. The filtrate is boiled for a few moments to ensure the complete precipitation of $Cr(OH)_3$, or longer to ensure that of NiS, whose presence is indicated by a brown or nearly black colour of the filtrate. Finally it is directed to wash with water containing a little $(NH_4)_2S$, and to keep the filter covered, in order to avoid the oxidation of the sulphides by the air, by which soluble sulphates may be formed.

2. Under the conditions of the procedure, which provides for a small excess of NH_4OH in the presence of NH_4Cl, aluminium, chromium, iron, titanium, zirconium, and beryllium are completely precipitated as hydroxides and uranium as ammonium diuranate, $(NH_4)_2U_2O_7$. All of these precipitates are white except those of chromium, uranium, and iron; $Cr(OH)_3$ is greyish green, and $(NH_4)_2U_2O_7$ is yellow. The colour of the precipitated hydroxide of iron varies with the state of oxidation of the iron, pure ferrous salts yielding a white precipitate, and ferric salts a reddish brown one, while mixtures of them yield green or black precipitates. In the alkaline mixtures the precipitate is rapidly oxidised by the oxygen of the air, and undergoes corresponding changes in colour. Manganous salts are also oxidised rapidly, with the result that brown $Mn(OH)_3$ separates. Under the conditions of the procedure zinc and nickel, when present alone, are completely dissolved; the same is true of cobalt except when it is present in large quantity, but zinc is precipitated when chromium is also present. The ammoniacal solution of nickel is blue and that of cobalt of a reddish colour, which darkens rapidly on exposure to the air owing to oxidation. If a smaller excess of NH_4OH is used than is directed, some zinc hydroxide, as well as cobalt hydroxide, may remain undissolved when large amounts of these elements are present, but this has no effect on the subsequent analysis. If, however, a much larger excess of NH_4OH is employed, a few mgrms. of aluminium and chromium may be dissolved, the latter giving a pink coloured solution.

3. The presence of a considerable quantity of NH_4Cl, such as is formed by the neutralisation of the acid already in the solution, serves to prevent the precipitation of $Mg(OH)_2$, and of $Mn(OH)_2$, and also to lessen the amount of $Al(OH)_3$ dissolved by the NH_4OH.

4. The influence of an excess of the NH_4OH and of the presence of NH_4Cl on the solubilities of the various hydroxides is explained by the mass action-law and ionic theory as follows:—In order that any hydroxide, say, of the type MO_2H_2, may be precipitated, it is necessary that the product $C_M.C^s_{OH}$ of the concentrations of the ions M++ and OH− in the solution in question attain the value known as the solubility-product. This varies, of course, with the nature of the hydroxide, but for all the elements of the iron group and also for magnesium it has so small a value that, even in a solution containing in 50 cc. only 1 mgrm. of the element and a slight excess of NH_4OH, the product $C_MC^s_{OH}$ exceeds it, and precipitation results. When, however, much NH_4Cl is also present, this greatly reduces, in virtue of the common ion effect, the ionisation of the NH_4OH and therefore the OH− concentration in the solution, so that now for certain elements the product $C_MC^s_{OH}$ does not reach the solubility value, even when C_M is moderately large (say, 500 mgrms. in 50 cc.). This is true of magnesium and manganese; but in the cases of aluminium, chromium, and ferric iron the solubility of the hydroxides in water is so slight that even in NH_4Cl solution the solubility is not appreciable.

If these were the only effects involved, the greater the excess of NH_4OH added, the less would be the solubility of any hydroxide, but other influences come into play with certain of the elements. These influences are of two kinds. The first of these is shown by zinc, nickel, and cobalt. In the case of these elements, just as with silver and copper, the excess of ammonia combines with the simple cation M++ forming complex cations of the types $M(NH_3)_2$++ and $M(NH_3)_4$++, thereby removing the simple cation from the solution and making it necessary for more of the hydroxide to dissolve, in order to bring back the value of $C_MC^s_{OH}$ to that o the solubility product. In such a case, the presence of NH_4Cl increases the solubility still further, since it greatly decreases C_{OH} and slightly increases C_{NH_4OH} and C_{NH_3}, owing to the common ion effect on the ionisation of the NH_4OH. Chromium also forms similar ammonia complexes, but in much smaller proportion.

The second effect is exhibited in the case of AlO_3H_3. This hydroxide is a so-called amphoteric substance; *i.e.*, is one which behaves both as a base and as an acid in

* From the *Journal of the American Chemical Society*, xxx., No. 4.

TABLE VII.

Analysis of the Aluminium and Iron Groups for Common Elements.

To the filtrate from the H_2S precipitate add NH_4OH and $(NH_4)_2S$ (P. 51).

Precipitate: $Al(OH)_3$, $Cr(OH)_3$, ZnS, MnS, FeS, Fe_2S_3, NiS, CoS.—Dissolve in HCl or HCl+HNO_3, evaporate, add NaOH, Na_2O_2, and Na_2CO_3 (P. 52).

Filtrate: Alkaline earth and alkaline elements.

Filtrate : $NaAlO_2$, Na_2CrO_4, Na_2ZnO_2. — Acidify with HNO_3, add NH_4OH (P. 53).

Precipitate: $MnO(OH)_2$, $Fe(OH)_3$, $Co(OH)_3$, $Ni(OH)_2$, $Ni(OH)_3$, $[ZnO_2H_2]$.—Dissolve in HCl, evaporate, heat with HNO_3 and $KClO_3$ (P. 61).

Precipitate : $Al(OH)_3$. — Dissolve in HNO_3, add $Co(NO_3)_2$, evaporate, ignite (P. 54).

Blue Residue : $Al_2O_3.xCoO$.

Filtrate.—Add $HC_2H_3O_2$ and $BaCl_2$ (P. 55).

Precipitate : $BaCrO_4$. — Dissolve in dilute HNO_3, add ether and H_2O_2 (P. 56).

Blue Coloration of Ether Layer : H_3CrO_7.

Filtrate: Salt of Zn. — Pass in H_2S (P. 57).

White Precipitate: ZnS. — Dissolve in HNO_3, add $Co(NO_3)_2$, ignite (P. 57).

Green Residue : $ZnO.xCoO$.

Precipitate : MnO_2. Boil with HNO_3 and PbO_2 (P. 62).

Violet Colour : $HMnO_4$.

Solution.—Add NH_4OH (P. 64). (*a*).

Precipitate : $Fe(OH)_3$. Dissolve in HCl, add KSCN.

Red Solution : $Fe(SCN)_3$.

Filtrate : $Co(NH_3)x(NO_3)_2$, $Ni(NH_3)_4(NO_3)_2$, $Zn(NH_3)_4(NO_3)_2$.—Pass in H_2S (P. 66).

Precipitate : ZnS, CoS, NiS.—Treat with cold dilute HCl (P. 67).

Solution : $ZnCl_2$, $[CoCl_2]$, $[NiCl_2]$.— Add NaOH and Na_2O_2 (P. 67).

Residue : NiS, CoS.

Solution : Na_2ZnO_2. Add $HC_2H_3O_2$ and H_2S (P. 57).

White Precipitate : ZnS.

Precipitate : $Co(OH)_3$, $Ni(OH)_{2-3}$.

Dissolve, evaporate, add NaOH.

To one half add KNO_2 and $HC_2H_3O_2$ (P. 69).

Yellow Precipitate: $K_3Co(NO_2)_6$.

To other half add KCN, digest, add NaBrO (P. 70).

Black Precipitate: $Ni(OH)_3$.

(*a*) Test for phosphate at this point (P. 63); if present, follow Table IX.

TABLE VIII.—Analysis of the Aluminium Group for Common and Rare Elements.

Filtrate from Na_2O_2 *Treatment*: Na_2CrO_4, sodium peruranate, Na_3VO_4, Na_2ZnO_2, $NaAlO_2$, Na_2BeO_2.—Add HNO_3, dilute, add $NaHCO_3$, heat in a closed bottle (*P. 58*a*).

Filtrate: Salts of chromic, uranic, and vanadic acids.—Add HNO_3 and $Pb(NO_3)_2$ (*P. 58*b*).

Yellow Precipitate: $PbCrO_4$.

Filtrate: $UO_2(NO_3)_2$, H_3VO_4, $[Pb(NO_3)_2]$.—Pass in H_2S.

Precipitate: PbS.—Reject.

Filtrate.—Add bromine, neutralise, add $HC_2H_3O_2$ and Na_2HPO_4 (*P. 58*c*).

Precipitate: $UO_2NH_4PO_4$. Dissolve in HCl, evaporate, add NaCl and $K_4Fe(CN)_6$ (*P. 58*d*).

Brown Precipitate: $(UO_2)_2Fe(CN)_6$.

Filtrate: H_3VO_4. Add NH_4OH, saturate with H_2S (*P. 58*e*).

Red Solution: Sulphovanadate.—Acidify (P. 58*e*).

Black Precipitate: V_2S_4—5.

Precipitate; $ZnCO_3$, $Al(OH)_3$, basic $BeCO_3$.—Dissolve in HCl, add NH_4OH (*P. 58*f*).

Filtrate: $Zn(NH_3)_4Cl_2$. Add $HC_2H_3O_2$, pass in H_2S (P. 57).

White Precipitate: ZnS.

Precipitate: $Al(OH)_3$, $Be(OH)_2$.—Dissolve in HCl, add ether, saturate with HCl gas (*P. 58*g*).

White Precipitate: $AlCl_3,6H_2O$.

Filtrate.—Evaporate, add 10 per cent $NaHCO_3$ solution, boil, pass in H_2S (*P. 58*h*).

Filtrate.—Acidify, boil, add NH_4OH.

Precipitate: $Al(OH)_3$, Fe_2S_3. Reject.

White Precipitate: $Be(OH)_2$.(*a*).

(*a*) Filter, and test the precipitate for uranium (*P. 58*h*).

TABLE IX.—Analysis of the Iron Group for the Rare Elements and for the Common Elements in the presence of Phosphate.

Precipitate from Na_2O_2 *Treatment*: $MnO(OH)_2$, $Fe(OH)_3$, $TlOH)_3$, $ZrO(OH)_2$, $TiO(OH)_2$, $Co(OH)_3$, $Ni(OH)_2$, $Ni(OH)_3$, $[Zn(OH)_2]$; $CaCO_3$, $SrCO_3$, $BaCO_3$, $MgCO_3$; and phosphates of these elements.—Dissolve in HCl, evaporate, heat with HNO_3 and $KClO_3$ (P. 61).

Brown Precipitate: MnO_2.

Solution.—Test a portion for iron; to the remainder add NH_4OH till nearly alkaline, then $NH_4C_2H_3O_2$ and $FeCl_3$, dilute, and boil (P. 65). (*a*).

Precipitate: Basic acetates, hydroxides, and phosphates of Fe, Tl, Zr, Ti.—Dissolve in HCl (1·12), shake with ether (*P. 65*a*).

Ether Layer: $FeCl_3$, $TlCl_3$.—Evaporate, dissolve in dilute H_2SO_4, add KI and Na_2SO_3 (*P. 65*d*).

Solution: $FeSO_4$.

Yellow Precipitate: TlI.

Water Layer: $TiCl_4$, $ZrCl_4$.—Add H_2SO_4, evaporate, add H_2O_2 and Na_2HPO_4 (*P. 65*b*).

White Precipitate: $Zr(OH)PO_4$.

Orange-yellow Filtrate: TiO_3. — Add Na_2SO_3 (*P. 65*c*).

White Precipitate: $Ti(OH)PO_4$.

Solution: Salts of Co, Ni, Zn, Ca, Sr, Ba, Mg. Add NH_4OH, pass in H_2S (P. 66).

Precipitate: NiS, CoS, ZnS. (See Table VII.).

Filtrate: Salts of Ca, Sr, Ba, Mg.

(*a*) Test for phosphate at this (Point. 63); if found absent, make the solution alkaline with NH_4OH (omitting the addition of ammonium acetate and $FeCl_3$), and filter.

consequence of its being appreciably ionised both into $3(OH^-)+Al^{+++}$ and into $H^+ + AlO_3H_2^-$ (or AlO_2^- and H_2O). With the H arising from the latter form of ionisation, the OH^- coming from the excess of NH_4OH combines to form H_2O, thus causing more AlO_3H_3 to dissolve until the value of $C_{AlO_3}.C_H$ again attains that of the solubility-product. Since $C_{AlO_3}.C_H$ = const. in any solution saturated with AlO_3H_3, and since the equilibrium equations $C_H.C_{OH} = K_W$ and $C_{NH_4}C_{OH} = K_3C_{NH_4OH}$ must be satisfied, it follows from combination of these equations that C_{AlO_3}/C_{OH} = const., and that C_{Al_3O} = const. $\times C_{NH_4OH}/C_{NH_4}$. The first equation shows that the quantity of aluminium dissolved is proportional to the concentration of hydroxide-ion in the solution, and that therefore it would be much greater in a solution of a largely ionised base like NaOH than in that of a slightly ionised base like NH_4OH. The second equation shows that the solubility in solutions containing NH_4OH and NH_4Cl (or other ammonium salt) is proportional to the ratio of the concentration of the base to that of the salt; so that the presence of ammonium salts tends to neutralise the solvent action of an excess of the hydroxide. All these conclusions are in accordance with the facts. BeO_2H_2 behaves in the same way as AlO_3H_3, forming the cation Be^{++} and the anion $BeO_2^=$ or $HBeO_2^-$.

5. It follows from the statements in the preceding notes that if the NH_4OH produces no precipitate it proves the absence of as much as 1 mgrm. of aluminium, iron, beryllium, uranium, titanium, and zirconium, also of chromium, if the mixture is heated to boiling after the addition of NH_4OH. Care must be taken not to overlook a small precipitate which might otherwise escape detection on account of its transparency. The mixture should therefore be well shaken, and allowed to stand two or three minutes in order that the precipitate may collect in flocks. This treatment also oxidises the iron when present in small quantity, and thus enables it to be more readily detected; for its precipitation in the ferric state is more complete.

6. When phosphate is present, magnesium, calcium, strontium, barium, and manganese may be partially or even completely precipitated by NH_4OH. It is therefore necessary, when phosphate is present to provide for the detection of the alkaline earth elements in the analysis of this precipitate. The normal phosphates and the monohydrogen phosphates of these elements are difficultly soluble in water, but dissolve readily in acids, owing to the formation in solution of the much more soluble dihydrogen phosphates or of free phosphoric acid; for example, according to the equations:—

$$(Ca^{++})_3(PO_4\equiv)_2 + 4H^+ + Cl^- = 2Ca^{++} + (Cl^-)_2 + Ca^{++}(H_2PO_4^-)_2,$$

$$Ca^{++}(H_2PO_4^-)_2 + 2H^+ + Cl^- = Ca^{++}(Cl^-)_2 + H_3PO_4.$$

Upon the addition of sodium or ammonium hydroxide to such a solution the hydrogen ion in equilibrium with the $H_2PO_4^-$ and H_3PO_4 is removed, and these dissociate into $HPO_4^=$ and $PO_4\equiv$, thus causing precipitation of the corresponding salts. When other elements, like iron, forming more insoluble phosphates are also present, it is evident that they will combine with the phosphate radical, thus leaving the alkaline earth elements in solution. On the other hand, when a soluble carbonate, or a large excess of a strong base, is also present, the alkaline earth phosphates will be partially converted into carbonates or hydroxides, leaving phosphates in solution.

7. The presence of any other acid radical which forms with the alkaline earth elements salts soluble in dilute acids but insoluble in ammonia may also cause their precipitation at this point. Such radicles are fluoride, borate, oxalate, and hypovanadate. The fluoride will ordinarily have been removed in the evaporation with acids in the preparation of the solution. The borates of the alkaline earth elements, though somewhat difficultly soluble, are not sufficiently so to cause them to be precipitated, except when present in very large quantity. Oxalate and hypovanadate, even if present, do not make any change necessary in the usual process of analysis; for, in the course of it, vanadate and much of the oxalate are separated from the alkaline earths in P. 52, and the remainder of the oxalate is destroyed in P. 61.

*8. Vanadium when present alone in moderate quantity may remain in solution, but when present in large quantity is partially precipitated by NH_4OH as a dark grey hydroxide, $VO(OH)_2$. This compound corresponds to the state of oxidation (oxide VO_2) to which vanadic acid (oxide V_2O_5) is partially or completely reduced by H_2S. It is an amphoteric substance which forms with acids soluble blue salts such as $VOCl_2$, vanadyl dichloride, and $VOSO_4$, vanadyl sulphate, and with bases hypovanadates such as $Na_2V_2O_5$ and $Na_2V_4O_7$. When other elements of this group are also present the vanadium may be completely precipitated with them, since their hypovanadates and vanadates are, in general, difficultly soluble substances.

*9. When a base is added to a uranyl salt (for example, UO_2Cl_2), uranyl hydroxide, $UO_2(OH)_2$, is first formed, but this is an amphoteric substance, and it is converted by the excess of base into salts of diuranic acid, $H_2U_2O_7$, of which even the alkali salts are insoluble.

10. $(NH_4)_2S$ precipitates ZnS, MnS, NiS, CoS, and Tl_2S, and converts $Fe(OH)_2$ into FeS, $Fe(OH)_3$ into Fe_2S_3, and $(NH_4)_2U_2O_7$ very slowly into UO_2S. The hydroxides of aluminium, chromium, titanium, zirconium, and beryllium are not affected by the $(NH_4)_2S$. Whether a hydroxide precipitate is converted into a sulphide precipitate or whether the reverse reaction takes place depends on the relative solubilities of the two compounds and on the relative concentrations of sulphide-ion and hydroxide-ion in the solution. Since, in the solution the concentration of the sulphide-ion greatly exceeds that of the hydroxide-ion, even difficultly soluble hydroxides would be converted into more soluble sulphides, provided that the difference in solubility were not too great. In the case of hydroxides, like $Al(OH)_3$, which are not so changed, their sulphides are so much more soluble that they do not form in aqueous sulphide solutions.

11. The sulphides of iron, nickel, and cobalt are black, Tl_2S and UO_2S are dark brown, ZnS is white, and MnS is flesh coloured, but turns brown on standing in the air owing to oxidation to hydrated Mn_2O_3.

12. When nickel is present alone, or in large proportion in the $(NH_4)_2S$ precipitate, several mgrms. of it usually pass into the filtrate, making it dark coloured, and some NiS also passes through the filter with the wash water. In this case it is useless to try to remove the NiS by filtering again, but it can be coagulated by boiling for several minutes. This behaviour of nickel, as stated above, can be avoided altogether by passing H_2S into the NH_4OH solution to precipitate the sulphide, instead of adding $(NH_4)_2S$. The formation of this brown solution depends upon the presence of ammonium polysulphide, for nickel may be completely precipitated by ammonium monosulphide in the absence of air. The nature of the brown solution is not known.

(To be continued).

Crystalline Compound of Magnesium Methyl Iodide with Amyl Ether.—Th. Zerewitinoff.—To prepare this crystalline compound magnesium ribbon, dry methyl iodide, dry amyl ether, and iodine crystals are allowed to act upon one another. When the action has ceased the mixture is heated with an upright condenser. To the saturated CH_3MgI solution thus prepared paraffin is added, and the crystals of $CH_3MgI(C_5H_{11})_2O$ separate after standing for some days.—*Berichte*, xli., No. 10.

NOTICES OF BOOKS.

The Scientific Foundations of Analytical Chemistry. By WILHELM OSTWALD. Translated by GEORGE M'GOWAN. Third English Edition. London: Macmillan and Co., Ltd. 1908.

THE third English edition of this work has been prepared from the fourth German edition, which appeared in 1904. Prof. Ostwald has himself revised the proof-sheets, and a few minor alterations and additions have been made. The excellent philosophical treatment of elementary analytical chemistry given in the book entirely does away with both the spirit and the letter of the now happily obsolete mechanical methods of identifying and estimating the commoner ions, and a perfectly new character is given to practical work. The general principles of physical and chemical methods of separating different substances are treated in the first part, and the applications of them to special cases are discussed in the second, in which the metals are classed together in the usual analytical groups. An appendix describes some simple experiments, which can easily be performed, to illustrate the general principles which underlie analytical chemistry.

A Treatise on Qualitative Analysis and Practical Chemistry. By FRANK CLOWES, D.Sc. (Lond.), F.I.C. Eighth Edition. London: J. and A. Churchill. 1908.

IT is perhaps unnecessary to call attention to the special features of a work which has reached its eighth edition, and which is as widely known as this for its admirably clear and concise directions for experimental work, both preparations and analysis. The book contains material for fairly advanced work, and the requirements of technical students as regards pure analysis have been taken into account, although no technical analyses are described. The section on the preparation and properties of gases has been revised, and in addition the detection of organic substances is treated rather more fully in this than in the previous editions.

Kelly's Directory of Chemists and Druggists. London: Kelly's Directories, Ltd. 1908.

THIS Directory contains lists of pharmaceutical, manufacturing, and analytical chemists, druggists, &c., alphabetically arranged according to the counties in which they reside, and also classified lists of trades and professions connected with chemistry and pharmacy both in London and the suburbs and in the counties. The names and addresses of veterinary surgeons are included, and the most important hospitals and dispensaries are also given. The book is well arranged and indexed, and special care has been taken to make it accurate and trustworthy in every respect.

Urbanora, the World's Educator. London: The Charles Urban Trading Co., Ltd. 1908.

THE use of the bioscope as an educational instrument opens up vast possibilities, both in elementary and advanced instruction, provided that the films are really good for their purposes and are not selected merely for spectacular effect. The Urbanora Company has engaged experts to obtain moving pictures of subjects of interest in Nature study, geography, history, surgery, &c., and although it is impossible to express an opinion upon the value of the films from a printed list, the photographs appear to have been chosen carefully, and the pictures should fulfil their special mission of exciting the interest of the student. In geography and history they aid the imagination to form clear conceptions of unfamiliar places and occurrences; in natural science they provide unique opportunities of making observations, especially of animal life; and in surgery they enable operations to be followed in every detail by large numbers of students simultaneously, and materially assist the lecturer in making his subject clear.

Research Work on Indigo. By W. POPPLEWELL BLOXAM, B.Sc. (Lond.), F.C.S., F.I.C. Published by Order of His Majesty's Secretary of State for India in Council. 1908.

THIS book contains reprints of papers published in various scientific journals by the workers attached to the University of Leeds, who have been engaged during the years 1905 to 1907 in investigating the chemistry of indigo. The chief outcome of their experiments is the perfecting of the tetrasulphonate process of analysing indigo, and the reports on the process are here given in full together with the discussions which took place at the meetings before which the papers were read; these discussions are an interesting part of the volume, and often throw light upon difficulties or disputed points. In the summary of the work which has been done the author is able to make the gratifying announcement that he and his collaborators have been able to perform practically all the experimental work originally planned, and proposals are now made which, if followed, should help the indigo grower to recover to some extent from the effects of the competition of the synthetic substance. Definite suggestions as to details of the manufacture, and more especially of the analysis of the leaf and finished product, are made, and thus the future of the industry is practically left in the hands of the planter and of those whose duty it is to provide funds to endow further research, of which there is urgent need.

The San Francisco Earthquake and Fire of April 18, 1906. Reports by GROVE KARL GILBERT, RICHARD LEWIS HUMPHREY, JOHN STEPHEN SEWELL, and FRANK SOULÉ. With a Preface by JOSEPH AUSTIN HOLMES. Washington: Government Printing Office. 1907.

THE reports of the experts who inquired into the effects of the San Francisco earthquake and fire of 1906 are contained in this book. Although the three engineers acted perfectly independently, there is a remarkable unanimity about the conclusions they have drawn with regard to the structure and materials of buildings which are most likely to withstand the effects of seismic disturbances, and these conclusions should be carefully studied by civil engineers. The general results of the investigations are summarised, and every effort is made to emphasise the lessons which are to be learnt from both the earthquake and the fire which followed it. Many instructive illustrations reproduced from the original photographs taken by the investigators show very clearly what type of structure was most liable to destruction, and the effects of the earthquake on the surface of the land are illustrated. In addition plans show the direction of the fault, the formation of which was responsible for the disturbance, although the earthquake, as a natural phenomenon from a purely scientific point of view, is studied in brief outline only, as a fuller account is given of it elsewhere by the Californian commission appointed for the purpose.

Kolloidchemie und Photographie. ("Colloid Chemistry and Photography"). By Dr. LÜPPO-CRAMER. Dresden: Theodor Steinkopff. 1908.

THIS monograph contains a short introduction to the chemistry of colloids, in which the many isolated facts which have been observed are brought together, those which are of special importance from the point of view of photography being enlarged upon. The applications of colloid chemistry to photographic problems are next con-

sidered, and here the attempt is made to further the introduction of a more scientific spirit into photographic practice. The chemistry of colloids is put before photographers in such a way that its special interest for them is plainly visible, and at the same time the attention of chemists who are engaged upon the investigation of the colloidal state is called to the problems in photography which are waiting for solution. These are so numerous and of such fundamental importance, both in practice and theoretically, that their study cannot fail to provide work of great interest and results of permanent value. The other branches of physical chemistry have hitherto been more thoroughly investigated than photo-chemistry, but deficiencies in this respect are now rapidly being made up, and books such as this are valuable reading for students who are ready to undertake research and who want some aid in selecting a profitable subject for investigation.

Kolloides Silber und die Photohaloide. ("Colloid Silver and the Photo-haloids"). By Carey Lea. Translated into German by Dr. Lüppo-Cramer. Dresden: Theodor Steinkopff. 1908.

The discoveries of Carey Lea in the chemistry of photography were so valuable that they may almost be said to have opened up a new region in scientific photography, and this translation of his works will be welcomed by German chemists. The most important papers, which appeared in the *British Journal of Photography*, the *Photographic News*, and the *American Journal of Science*, are translated in full, and notes are added by the translator, giving some account of more recent work, and emphasising the special importance of Carey Lea's researches on the allotropic modifications, or, as we should call them now, the colloidal forms of silver, and upon the photo-salts regarded as adsorption compounds of the silver haloids with silver. A short biography of the author and appreciation of the value of his contributions to the chemistry of photography is added.

Beiträge zur Allgemeinen Kolloidchemie. ("Contributions to the General Chemistry of Colloids"). By Dr. B. Szilard. Dresden: Theodor Steinkopff. 1908.

In this short monograph an attempt is made to lay the foundation of a comprehensive system by which all the phenomena of the colloidal state can be linked up, and the laws and principles regulating them formulated. Although the author has shown discretion in bringing together his material, the outcome of his work is on the whole disappointing. It may be summarised in a few words: he has established the existence of both organic and inorganic substances which possess the properties of albuminous bodies. These substances give rise to complexes by reactions which are neither chemical nor catalytic, but differ essentially from both classes of reactions. The value of the monograph lies in the stimulative effect the study of it may have upon chemists who are engaged in the investigation of the phenomena of colloidal chemistry, and who may find in it suggestions for fresh research in new directions.

Transparent Clear Crystalline Liquids.—D. Vorländer.—The author has prepared double-refracting, crystalline liquids which are as clear as water and transparent when spread on a glass plate; they are viscous, but do not form threads, and under the microscope they appear free from any turbidity or matter in suspension. The clear crystalline liquids can be made turbid by any process which destroys the uniform arrangement of the molecules, and the author explains at some length the theory of molecular form to account for the phenomena of crystalline liquids.—*Berichte*, xli., No. 10.

CORRESPONDENCE.

THE POTENTIAL ENERGY OF THE ELEMENTS.

To the Editor of the Chemical News.

Sir,—May I be allowed to point out to Mr. Daniel J. Rankin that, although, if correct, his values for the potential energy of the elements would be of very great importance, they are valueless until the method employed in calculating them is explained and shown to be valid, for the question of their correctness depends upon the validity of the method of calculation, and also, of course, upon the data employed.—I am, &c.,

H. Stanley Redgrove.

The Polytechnic, Regent Street, W.,
July 17, 1908.

THE THERMO-CHEMICAL RELATIONS OF CARBON, HYDROGEN, AND OXYGEN.

To the Editor of the Chemical News.

Dear Sir,—In the Chemical News of Jan. 26th, 1906 (vol. xciii., p. 37), I drew attention to values of thermo-chemical equivalence for carbon, hydrogen, and oxygen, giving approximate agreement with some organic compounds, when applied to the heat of formation of these compounds as compared with those obtained by experiment.

I find that similar results are obtained by comparing these thermo-chemical data in the case of the compounds cited below:—

	Experiment. Cals.	Calculated. Cals.
Tri-methyl methane	42·450	42·180
Tetra-methyl-methane. ..	47·850	48·990
Di-isopropyl	61·080	55·800

—I am, &c.,

J. C. Thomlinson.

6, Villa View, Low Fell, Gateshead-on-Tyne,
July 13, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Academie des Sciences. Vol. cxlvi., No. 24, June 15, 1908.

Solubility of Silver Iodide in Ammonia.—H. Baubigny.—By the method of crystallisation the author has found that the solubility of this salt in ammonia is much less than has hitherto been believed. At 16° the solubility in ammonia of density 0·926 is roughly of the order of 1/6000.

Ammoniacal Arsenic Chloride.—MM. Besson and Rosset.—When dry ammonia gas acts on arsenic trichloride at −20°, a yellow solid is formed, with composition corresponding exactly to the formula $AsCl_3.4NH_3$, It sublimes *in vacuo* at 200°, the sublimate being a substance of formula $2AsCl_3.7NH_3$. The compound $AsCl_3.4NH_3$ dissolves in liquid ammonia, but the evaporation of the ammonia does not leave behind the original substance, and hence the ammonia does not behave like a simple solvent, but like a reagent.

Colloidal Barium Sulphate.—A. Recoura.—If barium sulphate is formed by double decomposition in glycerin, water can be added to dilute the glycerin without causing

the precipitation of the sulphate. If the barium sulphate is obtained by neutralising a solution of sulphuric acid with barium ethylate, the colloidal substance can be obtained free from any electrolyte. The stability of a colloidal solution of barium sulphate is much increased by the addition of barium chloride or nitrate, and many electrolytes have no action upon the colloidal solution when barium salts are present, although they coagulate the pure solution.

Constitution of Tetramethyldiaminobenzhydryl Methylene Compounds.—R. Fosse.—If Weil's conclusion that tetramethyldiaminobenzhydrol is a ketone is correct the author's hydryl methylene compounds must result from the replacement of one atom of oxygen by a divalent residue, $=C<^{X}_{Y}$. The only compound the hydrol could yield with a mono-substituted methylene would be formed by the condensation of one molecule of ketene with two molecules of alkyl methylene. This conclusion is not confirmed by experiment. The hydrol unites with one molecule of methyl methylacetylacetate and methyl methylacetylacetone, yielding new hydrylmonoalkyl methylene compounds. The author has studied the properties of some of these new compounds and of their derivatives.

Action of Alkalis on Mono and Dimethyl Arsenic Acids and their Iodine Substitution Products.—M. Auger.—When caustic soda acts on sodium methylarsenate the latter is decomposed quantitatively at 250—280°, according to the equation :—

$$CH_3.AsO_3Na_2 + NaOH = CH_4 + AsO_4Na_3.$$

The same reaction occurs when sodium cacodylate is used. Triiodoacetic acid is very readily decomposed by alkalis into iodoform and alkaline carbonate, $CI_3CO_2Na + NaOH = CI_3H + CO_3Na_2$. Similarly, sodium diiodomethylarsenate and tetraiodocacodylate are split up by aqueous alkalis, giving methylene iodide :—

$$CHI_2AsO_3Na_2 + NaOH = CH_2I_2 + AsO_4Na_3,$$
$$(CHI_2)_2AsO_3Na + 2NaOH = 2CH_2I_2 + AsO_4Na_3.$$

Thus the atom of arsenic is detached from the molecule more readily than the two atoms of iodine bound to the carbon. This explains the quantitative formation of methylene iodide when a boiling solution of trisodium arsenite acts on iodoform. The iodoform unites with the tribasic arsenite, giving diiodomethylarsenate, $CHI_3 + AsO_3Na_3 = CHI_2AsO_3Na_2 + NaI$. Then this is decomposed by the alkali giving methylene iodide.

Lactone of Dioxy-3.4-butyric Acid.—P. Carré.—To prepare this lactone the monochlorhydrin of glycerin is diluted with water and potassium cyanide is added. When all action has ceased, concentrated sulphuric acid is added, and the whole is allowed to stand over-night. The chlorides of ammonium and potassium formed are filtered off, the solution is evaporated to dryness, and the residue taken up with acetone. After the latter has been distilled off baryta is added, and the solution of barium dioxybutyrate is precipitated with alcohol. The dioxybutyric acid is obtained by decomposing with alcohol. It loses water at 100°, and at a higher temperature two fractions can be distilled off; one is the lactone of oxy-4-crotonic acid and the other the lactone of dioxy-3.4-butyric acid. The latter is a colourless liquid, which can be distilled *in vacuo* without undergoing decomposition.

Double Phosphate of Magnesium and Monomethylamine.—Maurice François.—A double phosphate of magnesium and monomethylamine of formula $PO_4 \leq^{Mg}_{NH_3.CH_3.6H_2O}$ is known. It has been obtained in the crystalline state by mixing solutions of magnesium sulphate and sodium phosphate with monomethylamine. The corresponding phosphates of di- and trimethylamine do not appear to exist. Quantin's method of separating mixtures of di- and trimethylamine and ammonia cannot be applied to the separation of ammonia and monomethylamine.

No. 25, June 22.

Heat of Neutralisation of Acetic and Benzoic Acids, by Aniline in a Benzene Medium.—Léo Vignon and M. Evieux.—When the heats of neutralisation of acetic and benzoic acids by aniline in benzene solution are determined, it is found that no reaction occurs between the acids and base, and no salt is formed, thought salt is formed when the two substances are brought together either in the dry state or in aqueous solution. Apparently the salts first formed are destroyed by being dissolved in benzene.

New Method of Separating Silica and Tungstic Anhyride.—Ed. Defacqz.—The mixture of silica and tungstic anhydride is heated to a red heat in a current of hydrogen. Between 600° and 900° silica is not reduced by hydrogen, nor has chlorine any action upon it, while at the same temperature hydrogen reduces tungstic anhydride to lower oxides, or even metal, and chlorine readily yields hexachloride or oxychloride. When the action is over the substance is heated in a glass tube to a red heat in a current of dry chlorine, and the volatile products are condensed. All the tungstic anhydride then volatilises either as hexachloride or oxytetrachloride. This is dissolved in aqueous ammonia, and the tungsten is determined in the solution in the usual way. The residue of silica is weighed after the chlorine has been expelled by heating in air or hydrogen. This method of separation can be applied to any mixture of compounds containing oxygen, if one of the compounds can be reduced by hydrogen, and the resulting substance yields a volatile chloride.

Constitution of Derivatives of Diphenylmethane and Preparation of Ortho-di-amino Compounds of the same Series.—H. Duval.—The constitution of monoacetyldiphenylmethane is easily established, for when oxidised by chromic acid in acetic solution it yields benzophenone-*p*-carbonic acid. To prove the formula of dinitrodiphenylmethane dicarbonic acid, *o-o*-dinitro-*p-p*-diaminodiphenylmethane is diazotised and treated with cuprous cyanide. The *p-p*-dinitrile is thus obtained, and on saponification it gives the *o-o*-dinitrodiphenylmethane *p-p*-dicarbonic acid identical with the product obtained by directly nitrating diphenylmethane *p-p*-dicarbonic acid. The oxidation of ethyl *o-o*-dinitrodiphenylmethane *p-p*-dicarbonate by chromic acid in acetic solution leads to the formation of ethyl *o-o*-dinitrobenzophenone *p-p*-dicarbonate, the same substance as that obtained by oxidising dinitro-*p-p*-diacetyldiphenylmethane by excess of hypobromite, and treating with alcohol and hydrochloric acid. When nitric acid acts on *p-p*-diacetyldiphenylmethane in sulphuric acid solution *o-o*-dinitrodiphenylmethane *p-p*-dicarbonic acid is obtained.

β-Alcohol α-α-Dialcoholated Ketones. Transposition by Dehydration.—E. E. Blaise and I. Herman.—The ketone, $CH_2OH—C(CH_3)_2—CO—C_2H_5$, is transposed by alkalis, giving the unsaturated ketone methovinylisopropyl ketone, $\begin{matrix}CH_3\\CH_2\end{matrix}\geq C—CO—CH(CH_3)_2$. This might be explained in two ways :—Either a migration of the alcohol group occurs, followed by dehydration, or else dehydration is first effected, and then the methyl group migrates. Since the dehydration of oxydivalylethyl ketone does not lead to the formation of methovinylisopropyl ketone, it follows that the former explanation is correct, transposition occurring first and then dehydration.

Berichte der Deutschen Chemischen Gesellschaft, Vol. xli., No. 10, 1908.

Liquid Crystals.—Th. Rotarski.—The author gives an account of the properties and preparation of some liquid crystalline compounds which have not been described before. These include dianisyltetylene, $(CH_3O.C_6H_4.CH:CH)_2$, methoxycinnamic acid, methylamidobenzalphenylhydrazine, diethylbenzidine, bisdiphenylmethylol-biphenyl,

$[(C_6H_5)_2C(OH)C_6H_4—]_2$, and methoxy-cinnamic aldazine. The first of these is particularly interesting, for it shows that in methoxycinnamic acid the anisotropic liquid phase is not due to carboxyl or hydroxyl groups, but to the group $CH_3O.C_6H_4.CH:CH$, which is common to both substances.

Stereoisomerism in Compounds with Asymmetric Carbon and Pentavalent Asymmetric Nitrogen.—M. Scholtz.—The addition of halogen alkyl to alkylated *d*-coniin gives two different addition products with different physical properties. This phenomenon is due to the asymmetry of the pentavalent nitrogen atom. The same stereoisomerism can be observed in conhydrin and *α*-methyl-*α'*-phenyl piperidine. On the other hand, the addition of halogen alkyl to optically active alkaloids (except alkylated coniin and conhydrin) gives only one ammonium compound. It is found that the existence of two stable forms in *α*-substituted piperidine derivatives is conditioned by the volume occupied by the *α*-substituent which gives stability to the atomic groups attached to the neighbouring nitrogen atom. *α*-Picoline, however, behaves differently from piperidine derivatives with larger *α*-substituents, for it gives only one addition product with alkyl halogen. This appears to be because the methyl group, whether it is directly united with the asymmetric nitrogen or is the *α*-substituent in the pyridine ring, has not sufficient influence to give rise to two stable isomers.

Reduction of Alkyl Nitrates to Nitrites by reducing Salts in Alkaline Solution.—A. Gutmann.—The action of alkalis or arsenites upon alkyl thiosulphuric acid salts yields sulphite and thioethylhydroperoxide :—

$$SO_3Na.SC_2H_5 + NaOH = SO_3Na_2 + C_2H_5.SOH.$$

$$SO_3Na.SC_2H_5 + As(ONa)_3 + NaOH = SO_3Na_2 + AsO(ONa)_3 + C_2H_5.SK.$$

This behaviour is due to the per-acid character of thiosulphuric acid. As nitric acid has a similar character it might be expected that the nitric acid esters would behave in the same way as the thiosulphuric acid salts towards alkaline reducing agents, such as arsenites, cyanides, and sulphides. This is found to be the case with the ethyl and isoamyl esters of nitric acid, which behave precisely as predicted, according to the equations—

$$NO_2.OC_2H_5 + NaOH = NO_2.Na + C_2H_5.OOH.$$

$$C_2H_5.OOH + As(ONa)_3 = C_2H_5OH + AsO(ONa)_3.$$

Preparation of Boron Nitride and of Calcium Boride.—Alfred Stock and Walter Holle.—Boron bromide, which can be obtained comparatively easily in the pure state, reacts with ammonia, giving ammonium bromide and borimide, $BBr_3 + 6NH_3 = B(NH_2)_3 + 3NH_4Br$. Borimide is converted on heating into boron nitride. It is best to allow the bromide to drop into excess of liquid ammonia, and special precautions have to be taken in order that the tube through which the bromide is introduced does not get stopped up when it reaches the atmosphere of ammonia. It was found that the boron obtained from magnesium and boron trioxide is not pure enough to be used for making the boron bromide for this synthesis, and the authors endeavoured to prepare the element by reducing boron trioxide with calcium. At a dark red heat, however, this reaction does not lead to the formation of boron, but of almost pure calcium boride.

Double Dissociation of Quaternary Ammonium Compounds and Synthesis of Iodoaceto-nitrile.—J. v. Braun.—Iodoaceto-nitrile in the cold yields the direct addition product with dimethylaniline, and this product splits up quantitatively on warming as shown by the equation—

$$2C_6H_5.N(CH_3)_2(CH_2CN).I = C_6H_5.N(CH_3)_3.I + I.CH_2.CN + C_6H_5.N(CH_3).CH_2.CN.$$

Evidently the addition product undergoes double decomposition into four substances—$C_6H_5.N(CH_2)_2 + I.CH_2.CN$, $C_6H_5N(CH_3)CH_2CN + ICH_3$, the first and fourth of which then unite. Iodoacetonitrile can conveniently be prepared from methylcyanomethyl aniline by warming it with methyl iodide, the equation—

$$C_6H_5.N(CH_3)_2 + I.CH_2.CN \rightleftarrows [C_6H_4.N(CH_3)_2(CH_2.CN.I] \rightleftarrows C_6H_5.N(CH_3).CH_2.CN + ICH_3$$

occurring towards the left when excess of methyl iodide is used. These decompositions with bromo and iodoacetonitrile are the first known instances of the decomposition of an ammonium halogen compound yielding a halogen alkyl. In all cases in which tertiary aromatic bases react easily with iodoacetonitrile the reaction can be used to attach the cyanated methyl group to the tertiary nitrogen.

Characterisation of Primary, Secondary, and Tertiary Bases with 1 . 5-Dibrompentane.—J. v. Braun.—When 1 . 5-dibrompentane acts on primary amines in which there is an open chain, a hydrated carbon ring, a heterocyclic ring, or a benzene ring not substituted in the *o*-position, tertiary piperidines are obtained—

$$(CH_2)_5Br_2 + 3H_2NR = (CH_2)_5{>}NR + 2NH_2R.BrH.$$

These piperidines have basic properties and can be obtained in a pure state by distillation. If the benzene ring contains one or two substituents in the ortho position to the amido group, pentamethylenediamine derivatives, $R.NH.(CH_2)_5.NH.R$, are formed. Secondary amines of the fatty series, piperidine, &c., give with dibrompentane only quaternary piperidinium compounds :—

$$(CH_2)_5Br_2 + 2HNR_2 = (CH_2)_5{>}NR_2Br + R_2NH.HBr.$$

Secondary aromatic bases yield the same substance as the principal product of the reactions, besides small quantities of tertiary pentamethylene diamine bases, $R_2N.(CH_2)_5.NR_2$. Tertiary bases give only diammonium bromides—

$$(CH_2)_5Br_2 + 2NR_3 = Br(R_3)N.(CH_2)_5.N(R_3)Br.$$

MISCELLANEOUS.

Institute of Chemistry.—*Pass List, July Examinations*, 1908.—Of twenty-four candidates who presented themselves for the Intermediate Examination in London and Glasgow, the following fifteen passed :—H. L. Allen; B. S. Evans, B.Sc. (Lond.); C. E. C. Ferrey; H. A. Goldsbrough; L. Goodban; T. R. Greenough, B.A. (Cantab.); D. Henville; G. Hogan; A. P. Imrie; G. E. Johnson, B.Sc. (Lond.); Osman Jones; H. T. Reeve; E. H. Shepherd, B.Sc. (Lond.); R. W. Warrick; and J. Young. Twenty candidates presented themselves for the Final Examination in the Branch of Mineral Chemistry, held in London and Glasgow, and fourteen passed :—J. Boyd; J. Brebner, M.A., B.Sc. (Aberdeen); R. Bruce; G. S. Butler, B.A. (Oxon.); A. M. Cameron, B.Sc. (Edin.); W. Cameron; G. A. M. Cunningham; M. C. Fletcher; G. A. Freak, B.Sc. (Lond.); J. V. J. Hayman, A.C.G.I.; A. Hepburn; J. W. H. Pattison; A. S. Petrie; and Miss M. M. J. Sutherland. In the Branch of Metallurgical Chemistry, of two examined in London, one passed :—R. T. Rolfe. One candidate was examined in the Branch of Physical Chemistry, but did not satisfy the Board. Of nine candidates who presented themselves in the Branch of Organic Chemistry in London and Glasgow, the following four passed :—N. C. Akers; J. Kenyon, B.Sc. (Lond.); H. R. Norton, B.Sc. (Lond.); and W. F. Russell. The Examination in the Chemistry of Food and Drugs, and of Water, was held in London, and of six who presented themselves for the Associateship in this Branch, five passed :—H. A. Caulkin, B.Sc. (Lond.); H. Heap, M.Sc. (Manc.); A. S. Joy, Assoc. R.C.Sc. (Lond.); C. J. H. Stock, B.Sc. (Dun.); and C. H. Warner. Three candidates were examined in this Branch for the Fellowship, and two passed :—K. S. Caldwell, B.Sc. (Wales), Ph. D. (Leipzig); and A. R. Tankard.

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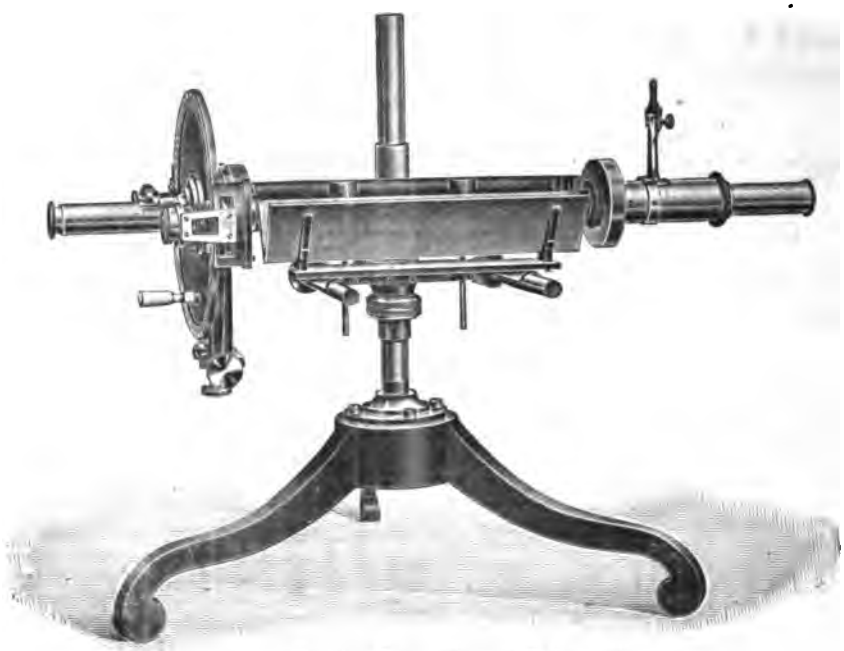

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THE CHEMICAL NEWS.

VOL. XCVIII., No. 2540.

NOTES ON LABORATORY APPARATUS.

By W. HEBER GREEN, D.Sc., University of Melbourne.

I. ON THE DETERMINATION OF DENSITY BY MEANS OF THE ORDINARY PEAR-SHAPED SPECIFIC GRAVITY BOTTLE.

SEVERAL authorities have objected to the use of this apparatus; thus, Ostwald, in "Physico-chemical Measurements," p. 90, says:—"An unpractical form of the pycnometer is in very general use. This is a small bottle with a ground-in and perforated stopper, its contents when the stopper is inserted forming the *supposed constant volume* in question. As the conical angle of the stopper must be small in order to get it to fit tight, a slight elastic deformation of the neck causes a considerable displacement of the stopper in the direction of its axis, which creates a by no means negligible source of error not easily to be avoided.

"To attain a more rapid equilibrium of temperature it is better to make the bottle cylindrical than spherical as usually happens."

Against this statement must be put the fact that in a number of determinations of the density of water by means of a pear-shaped specific gravity bottle of 25 cc. capacity, it was found that the maximum error liable to occur was about 0·00002, equal to 0·5 mgrm., and that even this was almost entirely due to variations in the buoyancy of the atmosphere.

But in order to obtain this accuracy certain precautions were found to be necessary. The ordinary specific gravity bottle can only be used for temperatures above that of the surrounding atmosphere; in order to take an observation it was filled with the solution, and immersed to the neck in a thermostat whose temperature did not vary by more than 0·04°, and it was found that expansion of the solution through the perforated stopper was complete in from ten to fifteen minutes. The bottle was then removed from the bath, wiped dry, placed in the balance case to cool for fifteen minutes, and weighed at once to prevent loss by evaporation.

In accurate work the buoyancy of air is an essential factor.

If W = apparent weight of water required to fill the bottle;
M = apparent weight of solution required to fill the bottle; and
d = true density; then—

$$= \frac{M}{W}\left(1 + \frac{0{\cdot}0012}{d} - 0{\cdot}0012\right).$$

Or the following amounts should be subtracted from the observed apparent densities:—

Approximate density of solution .	1·1	1·2	1·33
Correction for buoyancy of air ..	0·0001	0·0002	0·0003

The above formula is only approximate, and entirely disregards variations of atmospheric density, which often amount to over 5 per cent of the total, and are sufficient to nullify the correction for all but the most concentrated solutions usually worked with.

When required, the most practical and accurate method of correcting for the buoyancy of air would be to use as a counterpoise a similar but sealed glass vessel of the same external volume as the specific gravity bottle, and weighted with shot or mercury until slightly heavier than the latter when full of water. This should replace the all but useless brass counterpoise now so often supplied by the manufacturer.

Perhaps the most serious fault to be found with the ordinary laboratory instrument is that the stopper is only roughly ground in, for it is obvious that the more carefully it is fitted the less will the capacity of the bottle be affected by its rotation.

In two bottles that were employed by the writer the stoppers were carefully re-ground with fine and flour emery, and at the same time the capacities were adjusted so that at the temperatnre of experiment the apparent weight in grms. of the water contents was exactly twenty-five times the true density of water.

In the course of time the repeated wear on the ground surfaces of the stopper and neck slightly diminishes the capacity of the pycnometer.

If the same bottle be used at different temperatures then allowance must be made for the expansion of the glass.

An obvious refinement would be to make the bottle of Jena glass, or preferably of fused silica.

II. A SIMPLE BUT EXTREMELY SENSITIVE FORM OF THERMO REGULATOR.

In a piece of physico-chemical work carried out in this laboratory various patterns of thermo regulator were used

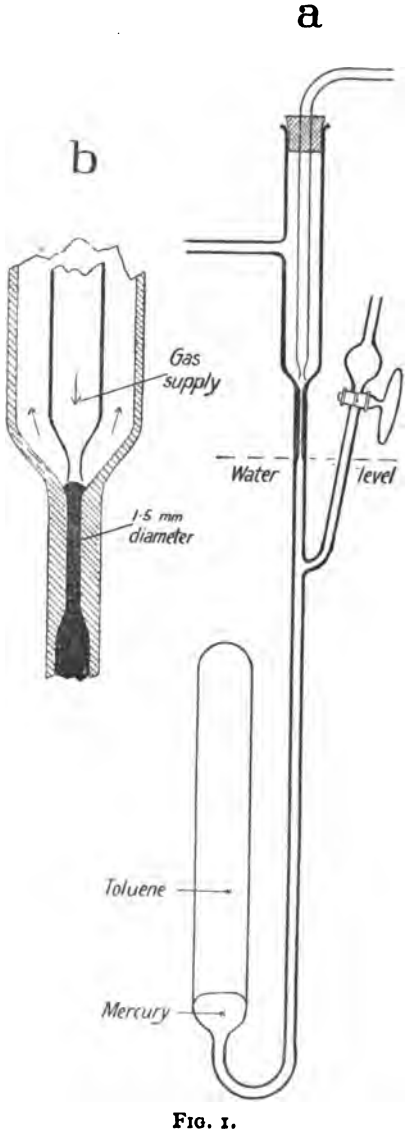

FIG. 1.

but finally a form was adopted, the most important novelty of which is that the orifice of the gas inlet tube is slightly opened out (see Fig. 1) instead of being left square, or cut off at an angle, or even constricted, as is often recommended.

In this form, as soon as the drop of mercury reaches and closes the funnel-shaped opening of the tube, its capillarity tends to force it down again and admit the gas supply; in the older forms the mercury will not enter and close the tube until an appreciable head of liquid has accumulated, and the required temperature exceeded, and will only be forced out again by the gas pressure with a rush when the temperature has fallen too low, and so an oscillatory instead of a steady temperature is apt to be maintained.

With this improved thermostat having a bulb of about 20 cc. filled with toluene as figured, the temperature was found to remain constant to at least within 0·01° for days and even weeks at a time, as long as the water in the bath was efficiently stirred, and this was provided for by means of a small propeller driven at high speed so as to cause a definite current up one side of the bath and down the other. A change from one temperature to another is readily effected by opening the tap of the thermostat, and, when the bath is at the desired temperature, adjusting the level of the mercury by suction applied through a rubber tube.

III. A Sensitive Temperature-compensated Barometer.

One drawback to the ordinary mercury barometer is that readings cannot be obtained accurate to 0·01 inch unless special precautions are observed and temperature corrections taken into account.

Water or glycerin barometers being about thirty feet high are sensitive, but unwieldy. Huyghens long ago suggested a combination of the mercury and glycerin barometers. In his form a narrow tube about a foot long was sealed on to the upper end of the wide tube containing the mercury; the glycerin was placed on top of the mercury and its upper free surface being in the small tube gave a sensitive indication of small movements of the mercury. By choosing tubes whose areas are as 1 : 30 a magnification of 6·28 could be obtained, but the temperature correction becomes increasingly important. With water instead of glycerin on top of the mercury, the changes of vapour pressure produce an error in the opposite direction, but of much greater magnitude.

After several experiments it was found that the vapour pressure of a paraffin oil (boiling-point about 230° C.), obtained by fractional distillation of kerosene, varied with the temperature at such a rate as almost exactly to neutralise the effects of the accompanying expansion of the mercury for the particular dimensions of tubes that were used.

A syphon barometer made in this way rises or falls one inch for a rise or fall of an eighth of an inch in the pressure of the air, and clearly indicates the momentary fluctuations of pressure inside the laboratory on a windy day.

The disadvantages of the instrument are:—

1. The difficulty of filling the tube and getting the liquid and top of the tube free from permanent gases. It is necessary to fill it from the upper end and advisable to leave a mercury trap into which any remaining gas can be forced after the apparatus is completed.
2. It is cumbersome, being over four feet long and not very portable.
3. It must be graduated by an arbitrary scale after comparison with a standard instrument.

Its advantages are:—

1. Great sensitiveness. A glance from a distance of several yards enables an observer to tell with certainty if the air pressure has altered by as little as one-hundredth of an inch during the progress of an experiment on, say, the boiling-point of a liquid or solution.

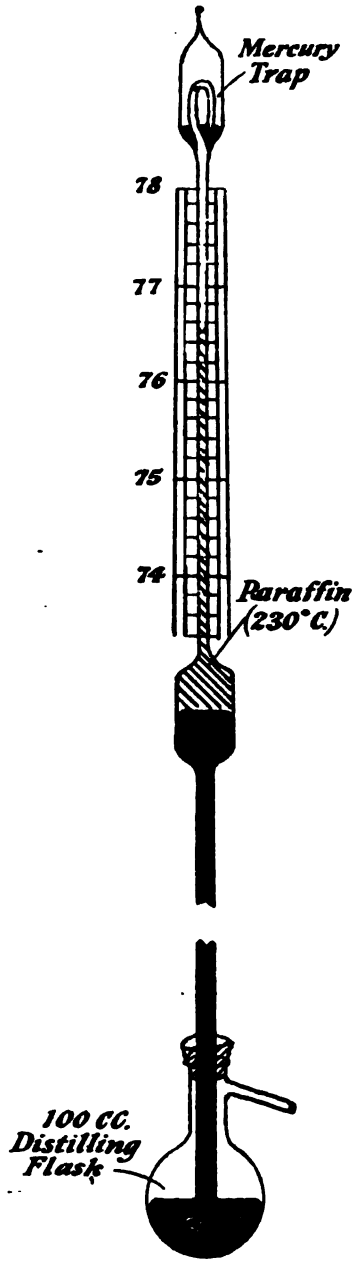

Fig. 2.—Sensitive Barometer.

2. The mercury surface in contact with the paraffin is always clean, and does not stick to the glass, and consequently responds instantly to minute changes of atmospheric pressure without tapping.
3. It is *automatically* compensated for changes of temperature.

About two years ago an English firm of scientific instrument makers advertised a sensitive temperature compensated barometer of a somewhat similar appearance, but gave no details of its construction or the principles on which it depended.

THE ESTIMATION OF GRAPHITE.

By FRANK BROWNE, F.I.C., Government Analyst, Hongkong.

WHEN graphite is heated at a bright red in an uncovered dish with ferric oxide several changes may be noted. The carbon entirely disappears, and the ferric oxide, though apparently unchanged at the surface, is darker in colour towards the bottom of the dish, owing to the formation of magnetic oxide. In order to see if the error due to this loss of oxygen could not be avoided, the action of heat and of various chemical reagents on ferric oxide has been studied, until finally it was found that heat alone could be so applied as to solve the difficulty.

If ferric oxide after being heated at a pale red in a *covered* crucible be then heated in the *uncovered* crucible at the same temperature, it slowly gains in weight, until after an hour's heating, provided always a fairly even temperature is maintained, the weight remains constant. If the heat be decreased from a pale to a dull red, then the oxide again increases in weight. Slight variations in temperature make practically no difference to the oxide, but it is essential in using the process hereinafter described that as even a temperature as possible should be maintained, and with a little practice this can be done without difficulty, and judging only by the eye. Based on the foregoing observations a process for the estimation of graphite has been devised. The ferric oxide to be used should be heated in a covered crucible at a pale red for two or three hours. The resulting oxide should be a slightly coherent magnetic powder, nearly black in colour. It will be found to be but very slightly hygroscopic. About 5 grms. of this oxide are taken, placed in a small uncovered shallow dish or wide mouth crucible, and heated at a temperature not exceeding a pale red for an hour, with occasional stirring, then weigh. Add about 0·5 of the graphite, stir together, and heat as before for one to two hours, stirring once or twice, and weigh. The carbon will be burnt off, and the oxide remains at the end in the same condition as at the commencement. When one estimation is finished another weight of graphite may be put into the same used oxide. A sample of graphite containing 9·15 per cent of mineral matter by potash fusion gave 8·98 and 8·96 per cent by this iron oxide method. Graphite freed from mineral matter showed by this process 99·73 per cent of carbon.

The mineral matter of coal may be determined in the same way, but the amount found will be about 0·5 per cent higher than as determined in the ordinary manner. This is what might be expected, as when coal is burnt to an ash the constituents are likely to react together, thus giving a figure somewhat lower than the true amount. Individual determinations, whether of graphite or coal, by this process will be found to agree closely; thus, using quantities from 0·5 to 1 grm. of coal, 9·21, 9·24, 9·34, and 9·35 per cent of mineral matter were indicated.

THE IODOMETRIC DETERMINATION OF ARSENIC AND ANTIMONY ASSOCIATED WITH COPPER.

By F. H. HEATH.

IT has been said that the iodometric process for the determination of copper can be used without error in the presence of arsenic and antimony provided that the latter elements are in the higher condition of oxidation. A. H. Low makes this statement and gives careful directions for the complete oxidation of the arsenic and antimony (*Journ. Am. Chem. Soc.*, xxiv., p. 1083). There appears, however, to be no record of any attempt to determine arsenic and antimony iodometrically after the separation of copper from a mixture containing the three elements.

It has been thought worth the while, therefore, to attempt the determination of arsenic and antimony in the filtrate from cuprous iodide after titration of the free iodine by means of sodium thiosulphate, and filtration. To this end the attempt has been made to reduce the arsenic and antimony by boiling with potassium iodide and sulphuric acid according to the method of Gooch and Gruener (*Am. Journ. Sci.*, Sept., 1891, xlii.) and determining these elements by reoxidation with a standard solution of iodine. The reactions involved in these processes may be expressed by the general symbols $M_2O_5+4HI = M_2O_3+H_2O+2I_2$, and $M_2O_3+4I+2K_2O = M_2O_5+4KI$.

The trials of the method were made with tartar emetic and potassium arseniate. The tartar emetic was dissolved in water and the antimony oxidised to the higher condition by means of standard iodine solution in presence of sodium or potassium bicarbonate. The amount of iodine required was taken as a measure of the amount of antimony used. The solution of antimony thus obtained, or the solution of arsenic taken in the higher condition of oxidation, was acidified and a known volume of a standard solution of copper nitrate was added. The copper was determined iodometrically with precautions previously recommended by Gooch and Heath (*Am. Journ. Sci.*, 1907, xxiv., 65).

A special precaution to be observed is the choice of the acid used during the determination of copper. Mineral acids must not be present on account of their tendency to bring about reduction of the arsenic and antimony by action of the excess of potassium iodide used in throwing out the copper. Such action causes high results on copper and low results on arsenic and antimony. A mixture of acetic acid and potassium iodide reduces the higher salts slowly. Tartaric acid is somewhat irregular in action and tends to cause an interfering precipitation of acid potassium tartrate. The action of citric acid is more favourable. The amount of iodine set free by the combined action of citric acid and potassium iodide upon an arsenic compound in the higher condition of oxidation is appreciable if the solution is allowed to stand, while in the case of an antimonic salt the action is not nearly so rapid.

A few determinations were carried out to test the action of citric acid quantitatively. Portions of tartar emetic were oxidised to the higher condition by iodine in presence of sodium bicarbonate, the solution was then acidified with citric acid, weighed amounts of citric acid and potassium iodide were added, and the solution was allowed to stand for a definite time to find whether there was any reduction. When the solution was acidified, after the oxidation with iodine solution, a little iodine was set free, and this was bleached with a drop of sodium thiosulphate before continuing the experiment. For the test with arsenic a standardised solution of potassium arseniate was used. The results of these experiments are shown in Table I.

From these results it appears that in the iodometric determination of copper associated with arsenic there must be no delay in titrating the iodine even when the free acid is citric acid; otherwise, some reduction of the arsenic acid and liberation of iodine may take place, thus affecting the result of the copper determination. Antimonic acid is not reduced appreciably, in a reasonable time, under similar conditions.

In the course of preliminary work the fact developed that tetrathionic acid, which results from titration of free iodine by sodium thiosulphate in the copper determination, makes trouble in the subsequent operation. For, when the solution is boiled, after addition of sulphuric acid, the tetrathionic acid decomposes to give hydrogen sulphide and free sulphur, and sulphides of antimony and arsenic may be precipitated. Various means were tried to oxidise the tetrathionic acid and its decomposition products before reducing the arsenic and antimony. Solid iodine and potassium nitrite were tried for this purpose, but not very successfully. Bromine water and, better, liquid bromine were used with success.

It was found that if liquid bromine was added to the cold solution in sufficient quantity to decompose all the excess of potassium iodide present, and the solution then boiled, there was very little subsequent trouble on account

TABLE I.—*Effects of Citric Acid and Potassium Iodide in Reducing Arsenic and Antimonic Salts.*

Sb taken. Grms.	As taken as KH_2AsO_4. Grm.	Citric acid used. Grm.	KI used. Grm.	Vol. of solution. Cc.	Time of standing. Minutes.	I found. Grm.
0·1209	—	3	4	60	10	No colour with starch.
0·0797	—	3	2	50	20	
0·1057	—	3	3	60	20	
					→ 60	Trace.
—	0·1238	2	3	50	10	0·0009
—	0·1238	2	4	60	10	0·0012
—	0·1238	3	3	50	10	0·0019
—	0·1238	3	5	75	10	0·0017
0·1211	0·1238	3	5	100	10	0·0010
0·1211	0·1238	3	5	100	10	0·0017

TABLE II.—*Copper and Arsenic.*

KI used. Grms.	Vol. at end of precipitation. Cc.	Cu taken. Grm.	Cu found. Grm.	Error in Cu. Grm.	Liquid bromine used. Cc.	KI used. Grms.	As taken. Grm.	As found. Grm.	Error in As. Grm.
3	50	0·0700	0·0700	—	1·0	2	0·1238	0·1231	−0·0007
3	50	0·0700	0·0693	−0·0007	1·0	2	—	0·1231	−0·0007
3	50	0·0875	0·0869	−0·0006	1·0	2	—	0·1239	+0·0001
4	50	0·0700	0·0698	−0·0002	0·6 0·4	1	—	0·1235	−0·0003
4	45	0·0700	0·0703	+0·0003	1·0	2	—	0·1247	+0·0009
4	50	0·0700	0·0700	—	1·0	2	—	0·1235	−0·0003
5	70	0·1400	0·1407	+0·0007	1·0 0·5	2	—	0·1233	+0·0005
5	50	0·0910	0·0907	−0·0003	1·0 0·5	2	—	0·1239	+0·0001
4	50	0·0700	0·0703	+0·0003	1·0	2	—	0·1237	−0·0001
4	50	0·0875	0·0879	+0·0004	1·0	2	—	0·1234	−0·0004
5	65	0·1400	0·1410	+0·0010	1·0	3	—	0·1239	+0·0001
3	30	0·0875	0·0860	−0·0015	0·8	2	0·0495	0·0493	−0·0002

TABLE III.—*Copper and Antimony.*

KI used. Grms.	Vol. at end of precipitation. Cc.	Cu taken. Grm.	Cu found. Grm.	Error in Cu. Grm.	Liquid bromine used. Cc.	KI used. Grms.	Sb taken. Grm.	Sb found. Grm.	Error in Sb. Grm.
4	85	0·0700	0·0703	+0·0003	1·0 0·5	2	0·1417	0·1421	+0·0004
4	80	0·0700	0·0701	+0·0001	1·0 0·5	2	0·1727	0·1725	−0·0002
4	80	0·0875	0·0869	−0·0006	1·0 0·5	2	0·1286	0·1289	+0·0003
5	80	0·0735	0·0739	+0·0004	1·0 0·5	2	0·1641	0·1645	+0·0004
5	90	0·1050	0·1050	—	1·0 0·5	2	0·1378	0·1372	−0·0006
4	75	0·0875	0·0874	−0·0001	1·0 0·5	2	0·1329	0·1326	−0·0003
6	115	0·0700	0·0707	+0·0007	1·3 0·5	2	0·2474	0·2477	+0·0003
8	120	0·1575	0·1571	−0·0004	1·5 0·5	2	0·1419	0·1413	−0·0006

of tetrathionic acid. To decompose the remainder of the potassium iodide used in the precipitation of the copper about one cubic centimetre of bromine was added. If, after boiling for a short time, the solution did not become clear, it was cooled, a little more bromine added, and the boiling repeated to ensure complete oxidation and to expel excess of bromine. It is well to concentrate the solution somewhat at this point so as to remove most of the bromine; otherwise, when potassium iodide is added to bring about reduction of the arsenic or antimonic salt, so much of it may be decomposed by the bromine that the reducing action may not be complete. Some free iodine remains in the solution after the reduction process, and there was found to be some difficulty in bleaching it with exactness in the hot solution. If the solution was cooled starch indicator could be used. Accordingly in the cooled solution the free iodine, in presence of starch indicator, was bleached by adding an excess of dilute sulphurous acid. The solution was then diluted to 100 cc. or more and iodine solution added to a faint colour. This pale blue colour was then just bleached by careful addition of dilute sulphurous acid from a pipette. The solution was neutralised by use of sodium or potassium bicarbonate, and the arsenic or antimony determined by titration with standard iodine solution in the usual way. Following are tables showing results obtained by this method.

The procedure for the determination of copper and arsenic or of copper and antimony may be outlined as follows:—To the solution containing the copper and also the arsenic or antimony in the higher condition of oxidation, add 1 grm. to 2 grms. of citric acid. To precipitate amounts of copper not exceeding 0·3 grm. in a volume of

TABLE IV. Copper, Arsenic and Antimony.

KI used. Grms.	Volume at end of precipitation. Cc.	Cu taken. Grm.	Cu found. Grm.	Error in Cu. Grm.	As taken as KH_2AsO_4. Grm.	I required by theory to oxidise As. Cc.	I used to oxidise Sb. Cc.	Sb taken as found by I used. Grm.	Br used. Cc.	KI used in reduction. Grm	I required by theory to oxidise As and Sb. Cc.	I used to oxidise As and Sb. Cc.	Grm.
—	—	—	—	—	0·1238	32·54	17·87	0·1088	1·0	2	50·41	50·06	−0·0043
—	—	—	—	—	0·0495	13·01	13·46	0·0820	1·0	2	26·47	47·70	−0·0030
—	—	—	—	—	0·1238	32·54	15·40	0·0938	0·5	2	47·94	44·90	−0·0053
—	—	—	—	—	0·0990	27·21	18·12	0·1054	1·0	2	45·33	30·00	−0·0020
5	90	0·0700	0·0718	+0·0018	0·0495	13·01	17·15	0·1044	1·0 0·5	2	30·16	30·00	−0·0009
5	90	0·0700	0·0710	+0·0010	0·0990	27·21	21·86	0·1272	1·0 0·5	2	49·07	49·00	−0·0009
(b) 5	95	0·1222	0·1223	+0·0001	0·0990	27·21	29·68	0·1727	1·5 0·3	2	56·89	56·23	−0·0081
9	115	0·0875	0·0868	−0·0007	0·0990	27·21	20·20	0·1176	2·0	3	47·41	46·90	−0·0063
6	110	0·1050	0·1067	+0·0017	0·0990	27·21	15·55	0·0905	1·3	2	42·76	42·64	−0·0015
(b) 7	100	0·1613	0·1611	−0·0002	0·0990	27·21	18·92	0·1101	1·5	2	46·13	45·90	−0·0028

(a) Weight of iodine × 0·50 = error calculated entirely on Sb; weight of iodine × 0·30 = error calculated entirely on As.
(b) Separate portions of Cu were weighed out.

50 cc. add 3 grms. of potassium iodide; in a volume of 100 cc. add 5 grms. of potassium iodide. Titrate the free iodine with sodium thiosulphate solution. The reactions involved in the copper determination are $2CuSO_4 + 4KI \rightarrow 2K_2SO_4 + Cu_2I_2 + I_2$ and $2I + 2Na_2S_2O_3 = 2NaI = Na_2S_4O_6$. Filter off the cuprous iodide on asbestos. To the filtrate add 1 cc. of liquid bromine and boil the solution in an Erlenmeyer flask, using a trap to prevent loss by spattering. If, after boiling for a short time and allowing the large amount of free iodine to volatilise, the solution does not become clear, cool it, add a little more bromine (0·5 cc.) and boil again. When the solution has become clear, concentrate it somewhat (to about 60 cc.) to expel excess of bromine. Dilute to about 100 cc., add 2 grms. of potassium iodide, and boil to a volume of 50 cc. Cool the solution, bleach the free iodine by adding sulphurous acid, using starch as indicator. Dilute to 100 cc., add iodine solution to colour, and just bleach by careful addition of dilute sulphurous acid from a pipette. Neutralise the solution with sodium or potassium bicarbonate and titrate the arsenic or antimony with standard iodine solution in the usual way.

From the results obtained it seems possible by this method to separate and determine copper and arsenic, or copper and antimony, with errors of only a few tenths of a milligrm. It is also possible to determine the sum of arsenic and antimony present with a fair degree of accuracy, and to separate and determine copper when associated with both arsenic and antimony. In the latter case the sum of the arsenic and antimony may also be determined, but the values here obtained for copper tend to come a little too high and those for arsenic and antimony a little too low.

In conclusion, the author wishes to express his appreciation of the kind assistance and many suggestions given by Professor F. A. Gooch during the course of these investigations.—*American Journal of Science*, xxv., p. 513.

RESEARCHES ON THE DENSITY OF GASES.*

By PHILIPPE A. GUYE.

THE present article contains a *résumé* of the results obtained during the course of three years' work on the exact density of gases. The work has been carried on in collaboration with Messrs. Jaquerod, Pintza, Davila, Gazarian, and Baume, and until now has been the subject of only isolated publication (Jaquerod and Pintza, *Comptes Rendus*, 1904, cxxxix., 129—SO_2 and O_2; Guye and Pintza, *Comptes Rendus*, 1904, cxxxix., 679; 1905, cxli., 51—N_2O, CO_2, and NH_3; Guye and Davila, *Comptes Rendus*, 1905, cxli., 826—NO; Guye and Gazarian, *Comptes Rendus*, 1906, cxliii., 1233—HCl; Baume, unpublished, 1907—SO_2). These have contributed to the problem of the physico-chemical determination of exact molecular weights, with a view to checking up the atomic weights. This problem has become one of great importance, and has been taken up by Leduc (*Recherches sur les Gaz*, Paris, Gauthier-Villars, in part; *Ann. Chim. Phys.*, 1897); Lord Rayleigh (*Proc. Roy. Soc.*, 1888, xliii., 353—H_2 and O_2; 1892, l., 449—H_2 and O_2; 1893, liii., 134—O_2, H_2, N_2 atmo. air; 1894, lv., 340—N_2; 1896, lix., 198—Ar, He; 1897, lxii., 204—CO, CO_2, N_2O; 1904, lxxiv., 181—N_2O; *Phil. Trans. Roy. Soc.*, 1905, cciv., A, 351) Rayleigh and Ramsay (*Phil. Trans. Roy. Soc.*, 1895, clxxxvi., A, 187), Morley (*Zeit. Phys. Chem.*, xx., 1—O_2; xxii., 1896, xxii., 2—H_2), Gray (*Journ. Chem. Soc.*, 1905, lxxxvii., 1601—N_2, O_2, NO_2; *Proc. Chem. Soc.*, 1907—HCl), as well as Perman and Davies (*Proc. Roy. Soc.*, 1906, lxxviii., A, 28). Details of the methods and the method of cal-

* Carried on during 1904, 1905, and 1906 in the Physical Chemistry Laboratory of the University of Geneva. From the *Journal of the American Chemical Society*, xxx., No. 16; translated by Helen Isham, from *Archives des Sciences Physiques et Naturelles*, xxiv., 32—62.

culating the results, will be given under the head of generalities; next a review of the results obtained at Geneva compared with those of other experimenters, and finally a summary of those values which appear to the author to be most reliable.

I. *Generalities.*

Methods.—Two general methods have been used in density determinations, that of the balloon and that of the volumeter.

The balloon method, the details for which have been worked out by Regnault (weighing the balloon empty and filled with gas, against a counterpoise of the same volume, reducing to weight *in vacuo*, &c.) has been used in recent work, taking into account the correction for the contraction of the empty balloon, and the correction for the compressibility of the gas (deviation from the law of Mariotte between the existing pressure and that of 760 mm. Hg). The recent work is also characterised by a gradual reduction of the capacity of the balloon. Morley worked with balloons of from 8 to 21 litres capacity. Rayleigh used one of about 1·8 litres capacity, Leduc one of about 2·3 litres. The work in this laboratory has led to a still further reduction of the capacity of the balloon, and two balloons, destined to act as checks upon each other, one 0·8 litre, the other about 0·4 litre in capacity, filled at 0°, and under the same pressure conditions have been used. (Experiments using still smaller balloons are being carried on at present).

Perman and Davies have used a balloon of 0·5 litre capacity, while Gray has reached the lowest unit, and in the determination of nitric oxide used one of about 0·267 litre. Contrary to what one might expect, *à priori*, the determinations with the small balloons are at least as concordant, among themselves, as those with balloons of larger volume. The experiments with the 2-litre balloons below, show, on the whole, a better agreement than those with balloons varying from 8 to 21 litres. This may be due either to the fact that the corrections for the original weights (for contraction, &c.) are smaller with the small balloons, or that the chances of accidental error, especially those due to the condition of the surface of the glass, are diminished.

The only objection which can be raised to the use of the small balloons is the possibility of the condensation of gas on the inner wall. If there were such condensation, the density of gases measured in small balloons should be greater than in the larger balloons, and this error should be especially great in the case of the hygroscopic gases, sulphur dioxide, ammonia, hydrochloric acid gas, &c.

The recent determinations show that while the capacity of the balloons varies to a great extent, the results obtained are very concordant, especially in the case of ammonia and sulphur dioxide.

By the volumeter method (capacity = about 1·8 litres) Perman and Davies found 0·77085 grm. for the weight of a litre of ammonia gas under standard conditions, while by the balloon method they obtained 0·77086 grm. for the same gas, using a balloon of 0·5 litre capacity. Guye and Pintza, using a volumeter of 3·5 litres capacity, found 0·7708. Thus there is no appreciable difference between the values obtained by the large and small balloons. Jaquerod and Pintza have obtained the value 2·9266 grms. for the weight of 1 litre of sulphur dioxide, using a volumeter of 3·5 litres capacity; Leduc, using a 2·3 litres balloon, found 2·9266; and Baume has recently repeated this determination, using two balloons of 0·53 litre and 0·32 litre capacity, respectively, on gas prepared under the same conditions as that used by Jaquerod and Pintza, and has found 2·9266 grms.

The only necessary precaution consists in rinsing the balloon, not only with air dried over phosphorus pentoxide, but also several times with the well-dried gas, the density of which is to be determined, taking care that between each rinsing operation the vacuum in the balloon is as perfect as possible, and that air does not enter the balloon between each determination.

It might nevertheless be assumed that there is a slight condensation of the gas on the inner wall of the glass, similar to the moisture adhering to glass. The author's experience (in collaboration with Gazarian) on the density of hydrochloric acid gas, which is extremely hygroscopic, does not justify this assumption. In fact, the balloons were at first rinsed only two or three times with dry hydrochloric acid gas. Under these conditions with a balloon of 0·385 litre capacity, the values for the density showed a regular decrease, and did not become constant until after the fourth determination. Using a larger balloon (0·818 litre capacity) this point was not reached after the seventh filling. These facts led to the conclusion that after twelve successive rinsings with the dry gas, each rinsing followed by a complete evacuation of the balloon, the condensation of the gas on the interior wall of the balloon is not sufficiently great to affect the results to an appreciable extent, provided, air has not entered the balloon during the process of rinsing. (In the case of non-hygroscopic gases 5 to 6 successive rinsings, each followed by complete evacuation, are sufficient). (If Bunsen's observations are correct, the moisture given off by the glass at high temperatures is not due to condensation on the surface, but to water incorporated chemically or physically in the mass of the glass. (See Note).

(Note.—If we admit that the condensation of the gas on the interior surface of the balloon is not generally appreciable, still we do not claim that it is absolutely nil. In order to determine the condensation most accurately, two or more balloons of very different capacities should be filled at the same time, under the same conditions of pressure, and with gas from the same source. The determinations of the densities of nitric oxide and sulphur dioxide, using two balloons filled simultaneously, furnish some preliminary information on this subject. The following averages have been found for the weight of a litre of these gases, determined simultaneously with two balloons:—

Weight of a Litre of NO (*G. and D.*).

No. of observation.	Capacity of balloon. Litre.	Weight of 1 litre. Grms.
7.	0·8	1·3401
7.	0·4	1·3403
5.	0·5	2·92666
4.	0·3	2·92659

It may be claimed that this condensation was not on the surface of the glass, but in the special grease used to insure a tight cock. It may be well to cite here the recent work of Swinton (Chemical News, 1907, xcv., 134), who has shown that the gases, hydrogen and helium, have no chemical action on the surface of glass, and that the surface condensation is of a purely mechanical nature. The recent observations of Travers (*Proc. Roy. Soc.*, 1906, lxxviii., A, 9) on the condensation of gases by solid bodies should not be overlooked, if the density of a gas is to be determined with an accurracy exceeding 1 part in 10,000).

The *volumeter method*, used as an exact method for the first time by Morley in the determination of the density of hydrogen has been used in two ways. According to the first method the gas is evolved from an apparatus which may be weighed, and constructed in such a fashion that the gas alone, in a state of perfect purity and dryness, escapes. The gas then passes through a tight-fitting joint into a system of one or more balloons of known capacity, maintained at 0°, and which have been evacuated by a mercury pump. The pressure is measured either with a special manometer or simply by connecting the apparatus with the tube of a barometer. Morley employed the latter method in his fourth series of measurements of hydrogen, and it has also been employed at Geneva. Knowing then the temperature and the pressure, the capacity of the balloons, and the loss of weight of the generating apparatus, the density may by calculated.

According to the second method the volumeter, previously evacuated, is filled with the pure gas. The process

of purifying may be more complete, as there is no limit to the weight of the apparatus used for that purpose. The volumeter being filled with the pure gas is held at 0°, and the exact pressure about (760 mm.) is read. Then the gas is absorbed in a suitable apparatus, previously evacuated and weighed, and connected to the volumeter by a tight-fitting joint.

The densities of hydrogen (Morley), oxygen, and sulphur dioxide (Jaquerod and Pintza) have been determined by the first method, by the second those of nitrous oxide, carbon dioxide, and ammonia (Guye and Pintza), and, more recently, ammonia (Perman and Davies).

The volumeter method used does not take into account the correction for the contraction of the balloons, but only that necessary for the reduction of the weights to weights *in vacuo* (for the evolution or absorption apparatus is weighed with a counterpoise of the same glass and the same volume), and finally the pressure coefficient of the gas in order to calculate the volume at a pressure of 760 mm.

Whichever method is employed (balloon or volumeter), the correction for the pressure coefficient of the gas is negligible, since the pressure differs from 760 mm. by only a few mm.

Choice of a Unit.—A common unit is indispensable. Leduc has reduced his values either to the density with regard to air, or to the weight of a litre of gas at Paris, or both. Morley expresses his results in weight per litre under standard conditions; that is, at 0° and 760 mm. pressure, at sea-level and latitude 45°. Rayleigh calculates his results either to density compared with air or compared with oxygen (taken equal to 32), or as the weight of 1 litre at London or at Paris. At Geneva the weight of the litre under standard conditions has been directly determined. Gray, as well as Perman and Davies, has likewise used this unit, which is the unit for all results which are given in the following pages.

D. Berthelot (*Comptes Rendus*, 1907, cxliv., 269) has recently calculated the greater number of measurements, comparing densities to that of oxygen. Instead of taking one value for oxygen, he adopts, in each case, the value obtained by each investigator for the density of this gas. This system presents several disadvantages:—First, in the case of those determinations made by investigators who have not determined the value for oxygen, the choice of such a value becomes somewhat arbitrary. Second, it might happen that the value for oxygen, even when determined by the investigator, was the most inaccurate of all his determinations; thus the accuracy of his other values would be diminished. This is, the case, for example, at Geneva, where the value for oxygen has been determined with less precision than for the other gases.

These disadvantages disappear when all results are calculated to the weight of 1 litre of the gas under standard conditions. All the investigators, with the exception of Leduc, have recorded the exact volume of the balloons employed. Leduc's values may be calculated, since he has determined the weight of a litre of air at Paris.

There is, finally, another reason for expressing the density by the weight per normal litre, and that is the fact that the volume of the balloon can be calibrated more accurately than the weight, of a gas for comparison, can be determined in the same balloon. Leduc reports that after an interval of two years the capacity of a balloon originally found to be 2·27636 litres, was 2·27630 litres, or the variation was 1:38,000. At Geneva an accuracy of 1 part in 30,000 to 35,000, between successive calibrations, has been easily realised. It is necessary to determine the capacity of the balloon for water at 0°, and under these conditions no correction for the expansion of the glass is required. (Difficulty was experienced in weighing the balloon filled with water at 0°, as the water expands when the balloon is placed on the balance. This has been overcome by attaching a bulb tube to the tubule of the balloon, by means of a ground joint or a rubber tubing, the whole apparatus having a counterpoise of the same glass and the same volume).

Choice of Final Values.—The following are some of the rules which have guided us in making a choice between the various values obtained for the density of each gas.

First, the nature of the method employed for the production and purification of the gas has been taken into consideration. This is, to our mind, the most important consideration in the density determinations. Those results showing concordance in the values obtained for the gas prepared in two or three different ways have therefore been given most weight.

A second consideration, equally important, is the concordance obtained by different investigators for the same gas. When the average values of two observers agree, while that of a third experimenter differs by a considerable amount, they have not been given equal weight, but the third value has been given less weight than the first two. Frequently, an experimenter has made a more or less accurate determination of the limit of error of his work. We have applied the correction in such a way as to bring the value nearer the mean of the other two observers. Similarly, when an error was discovered in the course of subsequent work, we have not taken into account that value known to be erroneous.

As will appear in the following discussion, while the preceding rules are suggested, an accurate choice is difficult to make. We would add that, in abandoning several of Leduc's values, giving preferences to others, generally more recent, in their stead, there has been no intention of diminishing the importance of Leduc's work. If the modern determinations have benefited by a more perfect technique, still we must recognise that to Leduc belongs the credit of having been the first to give a complete solution, theoretical and experimental, of the problem of the rigorous determination of the molecular weights of gases as a function of their densities.

(To be continued).

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

Part III.—Analysis of the Aluminium and Iron Groups, including Beryllium, Uranium, Vanadium, Titanium, Zirconium, and Thallium.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

Continued from p. 44)

Procedure and Notes (continued).

**Procedure* 51*a*.—To the filtrate from the $(NH_4)_2S$ precipitate (P. 51) add 5 cc. NH_4OH (0·90), and completely saturate the solution with H_2S. (Pink or violet-red colour, presence of *vanadium*).

If no red colour results, boil the solution for several minutes in a casserole to expel most of the NH_4OH and $(NH_4)_2S$, filter off the sulphur, and treat the filtrate by P. 71.

If the solution assumed a pink or red colour, acidify it with HCl (1·12), heat it to boiling, and filter. Boil the filtrate in a casserole to expel H_2S, add 0·5 to 5 cc. 10 per cent $FeCl_3$ solution, and NH_4OH (0·96) until the mixture after shaking smells of it, and filter. (Filtrate, P. 71). Heat the HCl precipitate (with the filter if necessary) with 5 to 10 cc. HNO_3 (1·20) until the black precipitate is dissolved, and filter. (If the red coloration produced by H_2S was slight, evaporate the filtrate to about 2 cc.). Add to the solution a few drops of 3 per cent H_2O_2. (Orange-yellow or orange-red colour, presence of *vanadium*). Dissolve the NH_4OH precipitate by pouring a small portion of HNO_3 (1·20) repeatedly through the filter, and test for

* From the *Journal of the American Chemical Society*, xxx., No. 4.

vanadium with H_2O_2 in the same way, first adding a little water to the HNO_3 solution if it has a red colour owing to the presence of much ferric nitrate.

Notes.

1. An ammoniacal solution of a vanadate or hypovanadate quickly becomes yellowish red when H_2S is led into it, and this colour slowly deepens as more H_2S is absorbed, but the characteristic brilliant violet-red colour is obtained only when the solution is completely saturated with H_2S. The presence of ammonium salts tends to prevent the formation of this red compound, but their influence is overcome by the addition of a large excess of NH_4OH. These facts make it probable that this red compound is an ammonium sulphovanadate, from which the sulphur is readily split off as SH^- ion, owing to hydrolysis. Under the conditions of procedure, 0·2 mgrm. V can be easily detected, the solution then having a pink colour.

2. Upon the addition of HCl the sulpho salt is immediately decomposed, with formation of a black precipitate of V_2S_4 or V_2S_5. This precipitation is far from complete under these conditions, only about half the vanadium being thrown down. More or less sulphur will also be precipitated, but the dark colour of the sulphide is apparent even when less than 0·5 mgrm. V is present. Acetic acid may be used instead of HCl, but the proportion of vanadium precipitated as sulphide is not much increased.

3. When a three- to fivefold excess of a ferric salt is present and NH_4OH is added, the remainder of the vanadium is precipitated (probably as hypovanadate) together with the $Fe(OH)_3$.

4. The confirmatory test for vanadium with H_2O_2 depends upon the formation of pervanadic acid, HVO_4. 0·1 to 0·2 mgrm. V may be detected in a volume of 5 cc., provided that this solution is strongly acid. A very large excess of H_2O_2 is to be avoided, for this decreases the intensity of the colour, and may even decolorise the solution completely, if very little acid is present, probably owing to the formation of a colourless compound of pervanadic acid with hydrogen peroxide.

5. Molybdenum, if not completely precipitated by H_2S in P. 21, and tungsten, if it passed into the original acid solution (owing to the presence of phosphate or arsenate) in P. 3, may also be present in the filtrate from the $(NH_4)_2S$ precipitate. Upon saturating with H_2S molybdenum also gives a deep red colour which would obscure the test for vanadium, and might be mistaken for it, but tungsten gives no colour. Upon acidification with HCl, tungsten divides between the precipitate and filtrate, but molybdenum is thrown down completely as black MoS_3. (If the cold solution is acidified with acetic acid, the sulphomolybdate remains undecomposed for some time, and the MoS_3 is not completely precipitated even on boiling). In the final confirmatory test with H_2O_2 tungsten gives no colour, and molybdenum, even when a large quantity is present, gives only a pure yellow colour, but not an orange or red one.

Procedure 52.—Transfer the $(NH_4)_2S$ precipitate (P. 51) with the filter if necessary to a casserole; add 5 to 20 cc. HCl (1·12), stir for a minute or two in the cold, and then boil the mixture for two or three minutes; if a black residue still remains, add a few drops HNO_3 (1·42), and boil again. Dilute with a little water, filter off the sulphur residue, and evaporate the filtrate to a small volume to remove the excess of acid.

*In order to detect rare earth elements, and to remove them if present, treat this solution by *P. 52*a*.

*If uranium and vanadium are to be tested for later by *P. 58*a—h*, evaporate twice with a little HNO_3 (1·42), to destroy HCl.

Dilute the solution to 10 or 20 cc., make alkaline with NaOH solution, avoiding a great excess; add 10 to 20 cc. more water if so large a precipitate separates that the mixture becomes almost gelatinous. Cool by placing the casserole in cold water, and add 0·5 to 3 grm. solid Na_2O_2, in small portions with constant stirring. Then add 5 cc. 10 per cent Na_2CO_3 solution; boil for two or three minutes to decompose the excess of Na_2O_2, cool, dilute with an equal volume of water, filter with the help of suction, and wash with hot water. (Precipitate, P. 61; filtrate, P. 53, or if uranium or vanadium is to be tested for, *P. 58*a*).

Notes.

1. All the hydroxides and all the sulphides, except NiS, CoS, usually dissolve readily in cold HCl. If therefore there is considerable black residue after adding the HCl, it shows the presence of nickel or cobalt (or possibly vanadium); a very small black residue may, however, be due to FeS enclosed within sulphur. The fact that there is no such dark coloured residue does not, however, prove that nickel and cobalt are absent, for a considerable quantity of them (even 5 mgrms.) may dissolve completely in the HCl when large quantities of other elements, especially iron, are also present.

2. The fact that NiS and CoS dissolve so much less readily in dilute acids than do the other sulphides of this group seems to be due not to a lesser solubility in water, but to an unusually slow rate of solution, for nickel and cobalt are not precipitated by H_2S, even from a much more weakly acid solution, and their sulphides obtained by precipitation with an alkaline sulphide continue to dissolve in dilute acids without reaching a limit determined by the concentration of the H_2S and the nickel-ion or cobalt-ion in the solution, as would be the case if the phenomena were that of the solubility of a difficultly soluble sulphide.

3. The $(NH_4)_2S$ precipitate is first treated with HCl, partly in order to furnish the indication just referred to of the presence of nickel or cobalt, but mainly because much more free sulphide and sulphate would be formed by oxidation if HNO_3 or *aqua regia* were used at the start. The presence of the sulphate in considerable quantity in the solution interferes with the subsequent test for chromate (with $Ba(NO_3)_2$ in P. 55 or with $Pb(NO_3)_2$ in *P. 58*b*). If NiS, CoS, or V_2S_5 is present in the residue, HNO_3 must, however, be subsequently added to ensure the solution of these sulphides. *The HCl is destroyed by evaporation with HNO_3, since chloride interferes with the test for chromate with $Pb(NO_3)_2$ in *P. 58*b*.

*4. If the $(NH_4)_2S$ precipitate be allowed to stand for a long time before treating it with acid, or if the mixture be heated for a long time after the precipitation, $TiO(OH)_2$ and $ZrO(OH)_2$ may remain in part undissolved even by the boiling concentrated acids, owing to the fact that the hydroxides at first precipitated become partially dehydrated, in which state they are very difficultly soluble. If this happens at this point or in dissolving hydroxide precipitates obtained later in the analysis, the residue may be dissolved in a little HF in a platinum dish, and the HF then expelled by evaporating two or three times with HNO_3 (or HCl) nearly to dryness.

5. By NaOH, iron, manganese, nickel, cobalt, *titanium, *zirconium, and *uranium are completely precipitated, and do not dissolve in moderate excess; while aluminium, chromium, zinc, *vanadium, and *beryllium remain in solution or dissolve when a sufficient excess is added, owing to the fact that their hydroxides are amphoteric substances (see P. 51, N. 3), and form with the NaOH soluble aluminate, $NaAlO_2$; chromite, $NaCrO_2$; zincate, Na_2ZnO_2; vanadate, Na_3VO_4; and beryllate, Na_2BeO_2. Thallium in the thallous state also remains in solution, since TlOH is a readily soluble substance. When zinc and chromium are simultaneously present, they are precipitated in the form of a double compound, $ZnCr_2O_4$. Chromium would also be completely precipitated, owing to hydrolysis and the formation of a less soluble solid hydroxide, if the NaOH solution were boiled before adding Na_2O_2. It will be observed that

NaOH precipitates manganese, nickel, and cobalt, while NH_4OH does not. This occurs with manganese because of the far greater concentration of hydroxide-ion in the NaOH solution, and with nickel and cobalt partly on this account and partly because there is no complex formation, as there is with NH_4OH. $Mn(OH)_2$ is white, but rapidly turns brown owing to oxidation to $Mn(OH)_3$; $Ni(OH)_2$ is light green, $Co(OH)_2$ is pink, but from cobalt solutions a blue basic salt is first precipitated in the cold. If a large excess of NaOH be added, a little $Co(OH)_2$ dissolves yielding a blue solution, doubtless forming a salt such as Na_2CoO_2. This is to be avoided since then the cobalt will not be completely oxidised and precipitated upon the subsequent addition of Na_2O_2.

6. By the addition of Na_2O_2, $Fe(OH)_2$ is changed to dark red $Fe(OH)_3$, $Mn(OH)_2$ to brown hydrated MnO_2, $Co(OH)_2$ to black $Co(OH)_3$, $Ni(OH)_2$ partially to black $Ni(OH)_3$, and thallium is precipitated as dark red $Tl(OH)_3$, all of which are insoluble in excess of NaOH and remain, together with $TiO(OH)_2$ and $ZrO(OH)_2$, in the precipitate. Chromium and uranium, which after the addition of NaOH are present as soluble sodium chromite or as insoluble sodium diuranate, $Na_2U_2O_7$, are converted by Na_2O_2 into chromate, Na_2CrO_4, and peruranate which are soluble compounds, and remain in solution, together with the zinc, beryllium, and vanadium, which are still present as zincate, beryllate, and vanadate. The separation is more satisfactory, especially in the case of uranium when a large amount of Na_2O_2 is used.

7. Even a cold solution of Na_2O_2 decomposes rapidly with evolution of oxygen, and this decomposition takes place with explosive violence when the solution is hot. The peroxide is therefore added in small portions to the cold solution. A steady evolution of gas continuing after the mixture has been well-stirred is an indication that sufficient peroxide has been added. The mixture is finally boiled in order to destroy the excess of Na_2O_2, and to cause the complete precipitation of titanium, some of which first passes into solution, probably as the pertitanate. The solution is cooled because $Tl(OH)_3$ is somewhat more soluble in the hot solution than in the cold one. Even in the cold when thallium is present alone about 0·5 mgrm. will usually pass into the filtrate, but precipitation is complete when elements of the iron group are also present. The solution is diluted before filtering in order to avoid the disintegration of the filter-paper; it is often advantageous to support the filter by folding it together with a small hardened filter.

8. The Na_2CO_2 is added to cause the complete precipitation of the alkaline earth elements, whose hydroxides, especially that of barium, are somewhat soluble even in the presence of NaOH. $ZnCO_3$, though insoluble in a dilute solution of Na_2CO_3 alone, dissolves when much NaOH is present, owing to the nearly complete conversion of the zinc-ion into zincate-ion by the reaction $Zn^{++} + 4OH^- = ZnO_2^= + 2H_2O$. The Na_2CO_3 also serves to decompose the chromates of the alkaline earth elements; if it is not added, chromium may remain in the precipitate and escape detection. It is unnecessary to add the Na_2CO_3 when the alkaline earth elements are known to be absent.

9. Phosphate or oxalate, if present, divides itself in this procedure between the precipitate and solution in a proportion which depends on the nature and quantities of the metallic elements. (See P. 51, N. 6). Their presence does not cause any of the elements to precipitate which would not otherwise do so, in spite of the slight solubility of aluminium, zinc, and beryllium phosphates. This is due to the fact that the cations of these elements (Al^{+++}, Zn^{++}, Be^{++}) are present in the NaOH solution only at an extremely small concentration, owing to their combination with the OH ion to form anions, (AlO_2^-, $ZnO_2^=$, $BeO_2^=$).

10. Even when less than 1 mgrm. Cr is present as chromate, it imparts a distinct yellow colour to the alkaline solution, so that when a colourless solution results, it proves the absence of this element. Uranium in small quantity also gives a yellow colour to the solution, which is noticeable with 1 or 2 mgrms.; a moderate amount (about 10 mgrms.) gives a red colour, which is intense with still larger quantities. Alkaline solutions containing vanadium are colourless after boiling.

11. This separation with NaOH, Na_2O_2, and Na_2CO_3 is a very satisfactory one, except in the case of zinc. This element, when present in small quantities, is completely carried down in the precipitate when much iron, nickel, or cobalt, or especially manganese, is present. Provision for the detection of zinc in the precipitate must therefore be made.

12. If Na_2O_2 is not available, sodium hypobromite, NaBrO (prepared by mixing NaOH and bromine, as described in P. 70, N. 3), may be used as the oxidising agent, but it is not quite so satisfactory as Na_2O_2, for it does not oxidise $Cr(OH)_3$ so readily, and it is apt to oxidise some of the manganese to $NaMnO_4$ (especially if there is not a sufficient excess of NaOH present).

**Procedure* 52*a*.—To detect rare earth elements, and to remove them, if present, transfer the acid solution (P. 52) to a platinum dish (after destroying HNO_3, if any has been added, by evaporating in porcelain once or twice with HCl, 1·20), and evaporate just to dryness, Add to the residue 45 per cent HF solution, little by little, until the residue has been dissolved, or until about 25 cc. have been added, stirring with a platinum rod after each addition, and finally boiling gently under a hood for a minute or two if there is still a residue. Collect the residue on a filter supported on a platinum ring or in a celluloid funnel; collect the filtrate in a platinum dish, wash the residue thoroughly with water.

To the filtrate add 3 to 5 cc. HCl (1·20), and evaporate it just to dryness; add a little HCl, and evaporate again, and repeat these operations a third time. Dissolve the residue in a little water, adding HCl if necessary; transfer to a casserole, and treat the solution by the last paragraph of P. 52, first evaporating with HNO_3 to destroy HCl if chromium, uranium, and vanadium are to be tested for by *P. 58*a*—*e*.

Transfer the residue insoluble in HF to a platinum crucible; if the filter has been added, ignite to destroy it. Add 2 to 5 cc. H_2SO_4 (1·20), heat until sulphuric acid fumes are given off, cool, add 10 to 20 cc. water, heat, and filter. Treat the precipitate (which can consist only of $CaSO_4$, $SrSO_4$, or $BaSO_4$) with concentrated Na_2CO_3 solution by P. 6. To the H_2SO_4 solution add ammonia until alkaline. (White precipitate, presence of *rare earth elements*). Filter, and test the filtrate for calcium and magnesium. Wash the precipitate thoroughly, dissolve it in a little HCl, evaporate just to dryness in a platinum dish, dissolve in a little water, and add 5 to 10 cc. HF. (White precipitate, presence of *rare earth elements*).

Notes.

1. Among the so-called rare earth elements are included thorium, cerium, lanthanum, praseodymium, neodymium, yttrium, ytterbium, and a number of other similar elements. These elements, like the alkali earth elements, are characterised by the insolubility of their fluorides, but differ from the alkali earth elements in that their hydroxides are precipitated by ammonia, and that their sulphates are not precipitated by sulphuric acid. It is upon these facts that the separation described in the above procedure is based. If there is no residue after the treatment with HF, it shows, of course, that the rare earth elements are absent; if, on the other hand, there is a residue, it does not necessarily show their presence, except when it is known that alkaline earth elements cannot be present. (See P. 51, N. 6 and 7). The further treatment described in the procedure serves to eliminate the latter.

2. Owing to the fact that aluminium and chromium fluorides are somewhat difficultly soluble in HF, care must be taken to use a rather large quantity of this acid

when a residue remains. It is also obvious that the fluoride residue must be thoroughly washed if the formation of a precipitate with ammonia is to be considered a conclusive indication of rare earth elements. The final treatment of the NH_4OH precipitate is recommended to eliminate the possibility of error from these sources.

3. The HF solution, which may contain all the elements of the aluminium and iron groups, is evaporated repeatedly with HCl to expel the HF, which is especially apt to be retained when titanium and zirconium are present.

(To be continued).

NOTICES OF BOOKS.

Synthetic Inorganic Chemistry. By ARTHUR A. BLANCHARD, Ph.D. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1908.

THE preparation of inorganic compounds of commercial importance is well treated in this little book, which has much to recommend it as an introduction to qualitative analysis. The principles and methods employed in synthetical chemistry are illustrated by well chosen examples, and a general discussion of the essential features of each preparation is followed by directions for the experimental work, which are invariably given separately; thus the student has no excuse either for losing sight of the theory of the reactions owing to the abundance of experimental details on the one hand, or for neglecting practical precautions on the other. Finally, questions bearing upon each preparation are propounded, most of them well selected, though perhaps a trifle difficult for the students for whose use the book is intended, and in addition there are sets of questions relating to the whole group of metals previously treated; in some cases these include simple supplementary practical problems, and these general questions make considerable demands upon the reasoning power and knowledge of the student. There can be no doubt about the value of working through such a course as that given in this book as an introduction to analysis, but it is perhaps debatable whether the average student could spare the time for it; it is not impossible, however, that the better grasp of the fundamental principles of analysis which he ought to acquire from a systematic study of the synthesis of compounds of each group of metals might actually save him time in the end.

Spectrum Analysis. By JOHN LANDAUER, Ll.D. Translated by J. BISHOP TINGLE, Ph.D., F.C.S. Second Edition. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1907.

THIS book gives an account of the use of the spectroscope and special spectroscopic instruments and appliances, and contains all that is required for ordinary analytical purposes; the spectra of the elements are tabulated and absorption spectra are also treated. Rowland's table of the wave-lengths of the Fraünhofer lines is reproduced in full, and some account is given of the spectra of celestial bodies other than the sun, of the aurora borealis, zodiacal light, lightning, &c. In the new edition the descriptions of some of the older instruments are replaced by those of more modern forms which have been found superior in practice, and also the measurements of spectra have been revised, the values being taken from Watts' "Tables of Spectra."

A Pocket Handbook of Minerals. By G. MONTAGUE BUTLER, E.M. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1908.

THIS book is intended to be used in field work, and also as a supplement to lectures on mineralogy. It lays special stress upon the identification of minerals by means of their physical properties, and the arrangement is such that the user should be able by help of the information given to decide the name of an unknown mineral without much possibility of doubt. Plenty of space is left for the insertion of additional notes upon the various minerals, and some illustrations are given. Simple chemical tests are also occasionally described, mostly as regards the behaviour of the mineral before the blowpipe and upon the addition of dilute acids, but from the aim of the book these are not given so much prominence as physical methods of identification. The minerals are arranged according to chemical composition, and some notes on their uses and value are added.

Les Nouveaux Livres Scientifiques et Industriels. ("New Scientific and Industrial Books"). Volume I. Years 1902 to 1907. Paris: H. Dunod and E. Pinat. 1908.

THIS well arranged bibliography will be a boon to French engineers, manufacturers, and scientific men generally. It gives first a general alphabetical list of books published in the French language during the years 1902—1907 dealing with technical and scientific subjects. This complete index to the whole volume is followed by an alphabetical list of authors, while the bibliography proper occupies the greater portion of the book. It is divided into twenty parts, published quarterly, and each part is separately indexed. For every book the title, author's name, price, size, and number of pages are stated, together with a short *résumé* of the contents, and the catalogue thus gives every possible assistance to scientific men who wish to keep abreast of modern developments in any particular subject in which they are specially interested.

CORRESPONDENCE.

WOMEN AND THE CHEMICAL SOCIETY.

To the Editor of the Chemical News.

SIR,—As one of the signatories to the petition to the Council of the Chemical Society, I regret that the issue of a letter dated from Oxford, July 1st, 1908, by eleven of our most distinguished chemists, in which the petition is stated to be a declaration of opinion, renders it necessary to point out that it was signed by myself, and I believe many others, as a petition to the Council to ascertain the wishes of the Society on the question, and not as a petition for the admission of women.

I regret still more that the perfectly natural choice of the Society's printers for the printing of the letter and petition has led to the use of the same paper and type as the Council's circular, and of the official address labels, and has therefore doubtless in many cases caused the impression, until this was corrected by the Officers of the Society, that the Oxford letter signed by so many Past-Presidents and the petition were officially issued to show how great was the support.

Since signing the petition I have given much consideration to the question, and greatly sympathise with those women chemists who are debarred from the privileges of the Fellowship, and if there were nothing beyond the mere question of allowing them to vote, to use the Library, and to receive the Journal, I should unhesitatingly do all I could to further their admission.

Unfortunately there is a very grave issue beyond this, and as it is not mentioned in the very fair and impartial statement of the arguments on both sides issued by the Council, I venture to outline it.

Women being the weaker—I do not say inferior—sex have many privileges of courtesy, and if they are granted the full privileges of the Fellowship will have an advantage over men. I can perhaps make my meaning clearer by an example:—An unknown woman reading her first

paper before the Society will be listened to with an attention which would be denied to an unknown man, and more consideration would be given to it by the Publication Committee, and the reading and publication of the first paper is often an important step in a chemist's career. In this way the woman would have an advantage over the man, and might eventually gain an appointment, not upon merit alone, and displace a man; as a man generally supports a woman who has taken up the profession for which Nature has best fitted her—that of maternity—the woman chemist would in the long run displace a man, another woman, and their children. When I have to consider the interests of women, there is only one woman who really counts—my wife—and seeing that by voting for the admission of women to the Fellowship I may possibly be furthering the interests of another woman to the detriment of those of my wife, my sympathy for women chemists is greatly weakened.

The position of the distinguished chemists who signed the Oxford letter is such that they could not be displaced by any one, and consequently the above view has never probably occurred to them, and it is doubtless only a feeling of chivalry that has led them to issue their circular independently of the Council of which they are members, but I venture to think that the humble rank and file of the profession like myself will view the subject in a different light, and fear that the same feeling of chivalry will induce the leaders of the profession to give undue advantages to women should they be admitted as Fellows.

As after careful consideration of the whole question, I have now become an opponent of the admission of women, I would like to thank Dr. Divers for the admirable way in which he has given his side away; it requires little more testimony of the weakness of his case when a confession is made that the issue of a statement of the arguments for and against the admission of women is an unfair advantage to the opponents of the measure; when the omission of the obvious fact that the supporters of admission of women think that the time has now come for that to be accomplished is called still more unfair advantage to the opponents, the position becomes ludicrous; and when the Council, after presumably a full discussion of the subject, is supposed to have no official cognisance of the nature of the arguments against the admission of women, and the Secretaries are blamed when by order of the Council they issue a circular containing what is obviously an abstract of the discussion, it appears that what the supporters of the measure most dread is a full statement of the case on both sides being placed before the Fellows for their unbiassed consideration.

I would appeal to Fellows not to be influenced by the support that our most illustrious chemists have given to the movement, as their very eminence prevents not only their being able to view the whole subject, but also their interests being affected one way or the other, but to weigh carefully the arguments on both sides, and come to an independent decision.—I am, &c.,

H. Droop Richmond.

London, July 20, 1908.

EXPLOSIVE CRYSTALLISATION.

To the Editor of the Chemical News.

Sir,—Is not the true explanations of the mishaps described in issue of July 17 to be found in the fact that the vacuum was too great for the resistance to crushing of the glass receivers and plates, and that it was the pulsations of these yielding to continued over-exhaustion before they snapped that caused the bumping? Had the air-pump been a copper still or a vacuum pan they might have been flattened, *i.e.*, if they were as unequal to the strain as these receivers and plates prove themselves to have been. It was just as well the pressure was from outside to inside instead of from inside to outside. In the latter case the consequences might have been serious. It must be acknowledged that 19 mm. from an absolute vacuum is very nigh 15 lbs. to the square inch, so the total pressure on the outside of receiver and plate was several hundredweights. Are ordinary air-pumps made to stand such highly diminished internal pressure? I imagine not. Whether there was sudden absorption of heat at the moment of crystallisation, if crystallisation occurred *en masse* and suddenly cooled, the remaining volume of highly attenuated air can easily be ascertained by the physical chemistry of the substance used. But that for the moment I have no time to consider. In any case the pulsation causing bumping seems to debar the effects of sudden condensation.—I am, Sir, &c.,

John Geddes M'Intosh.

9, Parsonage Street, Cubitt Town, E.

POTENTIAL ENERGY OF THE ELEMENTS.

To the Editor of the Chemical News.

Sir,—I regret that through an error in transcribing, the potential given in my paper on "The Potential Energy of the Elements" (Chemical News, xcvii., p. 302), for tellurium, viz., 3228 should be 3288; a note should also have been appended stating that this was the potential of the theoretic element having a density 6·045 and atomic weight 124·57. The potential for tellurium, which I consider to be a compound of two elements, is 3137 K.

I would also remark that, in my view, tellurium cannot be split up into its components by ordinary chemical analysis, the energy reactions by this method being on too low a grade.

Tungsten is another compound element of an analogous nature to tellurium.—I am, &c.,

Daniel J. Rankin.

Orleans House, Peterborough, July 21, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Academie des Sciences. Vol. cxlvi., No. 26, June 29, 1908.

Decomposition of Alcohols under the Catalytic Action of Live Coals.—Georges Lemoine.—When live coals from a baker's oven are heated to redness and then allowed to act on alcohol at temperatures ranging from about 380° to 440° the decomposition of the alcohol is of the nature of a dehydrogenation, instead of dehydration, as with purified animal charcoal. Isopropyl alcohol, however, is an exception, and differs in its behaviour from normal propyl alcohol. This difference in the effect of animal charcoal and coal shows how different transformations can be effected by slightly different substances. The phenomena observed confirm the general law that catalysers lower the temperature of chemical transformations.

Action of Metallic Oxides on Primary Alcohols.—Paul Sabatier and A. Mailhe.—The authors have allowed the vapours of primary alcohols to act upon a number of different metallic oxides at temperatures between 350° and 400°. They find that the oxides can be divided into four classes according to their behaviour under these conditions: 1. Those which below 400° undergo no reduction and have no appreciable effect upon alcohols; these include calcium, barium, and strontium oxides, calcined magnesia, calcined silica, and titanic oxide TiO_2. 2. Those which are rapidly reduced to metal or lower oxide. 3. Those which are not reduced at temperatures below 400°, but act catalytically, yielding either aldehyde or ethylene hydrocarbon, or both. 4. Those which react only slowly and cause the condensation of the alcohol.

Preparation of Alkaline Chloroiridites.—M. Vèzes.—The author calls attention to the fact that the preparation of alkaline chloroiridites by reduction of the chloroiridates with neutral oxalate, described by M. Delépine (*Comptes Rendus*, June 15, 1908, cxlvi., 1267) is not new, but was reported seven years ago (*Procès-verbaux des Séances de la Société des Sciences Physiques et Naturelles de Bordeaux*, July 18, 1901, p. 112).

Molecular Weights of Phosphoric Acids determined by Cryoscopy.—H. Giran.—In acetic solution the molecular weight of metaphosphoric acid diminishes with the time, and tends towards a limit which is lower the more dilute the solution. The molecular weight of the meta acid is 400 and the formula is $(PO_3H)_5$. The same holds good for pyrophosphoric acid, which has the formula $(P_2O_7H_4)_3$ and the molecular weight 534. With the ortho acid the molecular weight varies with the time, all the results being included between the normal weight $PO_4H_3 = 98$ and the normal weight $(PO_4H_3)_2 = 196$. This might be interpreted by supposing that the limit is reached very rapidly. The molecular condensation of the ortho acid is the same in the liquid and solid state, and the condensation is certainly greater than 1. It is logical to conclude that the diminution of molecular condensation observed in passing from the meta to the pyro acid is continued when we pass from the pyro to the ortho acid. The last should then have a condensation lower than 3, and thus equal to 2. Thus solid or liquid orthophosphoric acid must be represented by the formula $(PO_4)H_3)_2$.

Tellurides of Arsenic and Bismuth. Cryoscopic Constant of Tellurium.—H. Pélabon.—Fused tellurium dissolves easily in arsenic. The liquid obtained is grey when little arsenic is present, and darkens gradually to black as the proportion of arsenic increases. The temperature of sodidification of the liquid decreases rapidly and regularly from 452°, the melting-point of tellurium, to 329°, the temperature of solidification of the mixture formed of 3 atoms of tellurium to 1 of arsenic. If the proportion of arsenic is increased the temperature of solidification rises, reaches a maximum value 362° for the compound As_2Te_3, then falls, becomes 355° for the mixture $9As + 11Te$, and again rises to 358° for all mixtures containing more than 1 atom of arsenic to 1 of tellurium. The fusion curve of mixtures of tellurium and bismuth from the melting-point of tellurium, 452°, has first an almost rectilinear part, which ends at 410°, corresponding to the eutectic mixture containing about 15 per cent of bismuth. From this point for mixtures richer in bismuth, the temperature of solidification increases, and reaches a maximum at 583° for the compound Bi_2Te_3. It then falls regularly to 263°, which is the solidification-point of the eutectic containing 1 per cent of tellurium. The temperature of solidification then increases to 270°, which is the melting-point of bismuth. Thus, if a small quantity of arsenic or bismuth is added to tellurium, compounds As_2Te_3 and Bi_2Te_3 are formed, and dissolve in the excess of tellurium. The study of the solidification of these solutions shows that the cryscopic constant of tellurium is 520.

Mechanism of Synthesis of Nitrogen Cycles.—L. J. Simon.—The action of pyruvic acid on paratoluidine gives a product,—

$$CH_3—\underset{\displaystyle NH—C_7H_7}{\overset{\displaystyle CO_2H}{\overset{|}{\underset{|}{C}}}}—CH_2—\underset{\displaystyle NH—C_7H_7}{C(OH)—CO_2H},$$

formed by the condensation of two molecules, $CH_3—\underset{\displaystyle NH—C_7H_7}{C(OH)—CO_2H}$. The action of water upon this substance may be represented by the two equations $C_{20}H_{24}N_2O_5 = C_{12}H_{11}NO + CO_2 + H_2O + H_2 + C_7H_7NH_2$, $C_{20}H_{24}N_2O_5 = C_{19}H_{22}N_2O_2 + CO_2 + H_2O$. The first substance formed is 2-6-dimethylquinoline carbonic acid. The second product is a cyclic compound formed by the removal of OH_2 and CO_2 from the COOH, NHC_7H_7, and CO_2H groups of the original condensation product.

Messinger and Vortmann's Method of Estimating Phenols.—J. Bougault.—The author has shown that Messinger and Vortmann's method of estimating phenols gives with salicylic acid a compound which has always the composition $(C_6H_2I_2O)_x$, and which is identical with Lautemann's "red substance." The formation of this substance can be utilised to separate and determine salicylic acid. The constitution of the red substance seems to be $\begin{vmatrix} C_6H_2I_2O \\ \\ C_6H_2I_2O \end{vmatrix}$, *i.e.*, it may be called tetraiododiphenylene-quinone.

Three New Primary Methods resulting from the Condensation of Sodium Benzylate with Propyl, Butyl, and Isoamyl Alcohols.—Marcel Guerbet.—Sodium benzylate reacts with propyl alcohol giving an alcohol of formula $C_{10}H_{14}O$. This is methylbenzylethyl alcohol, $C_6H_5—CH_2—CH(CH_3)—CH_2OH$, and the same reaction occurs with butyl and isoamyl alcohols:—

$$C_6H_5—CH_2ONa + C_4H_9OH = C_6H_5—CH_2—C_4H_8OH + NaOH,$$

$$C_6H_5—CH_2ONa + C_5H_{11}OH = C_6H_5—CH_2—C_5H_{10}OHNaOH.$$

The first alcohol is ethylbenzylethyl alcohol, $C_6H_5—CH_2—CH(C_2H_5)—CH_2OH$, and the second is probably isopropylbenzylethyl alcohol,—

$$C_6H_5—CH_2—CH(C_3H_7)—CH_2OH.$$

Researches on Diazo Compounds.—H. Duval.—The author has studied the action of nitrous acid upon the following orthodiamino compounds—of diphenylmethane *p*-*p*-dicarbonic acid, diphenylmethane *p*-*p*-dinitrile, *p*-*p*-diacetyldiphenylmethane, diphenylmethane, diacetyldiaminodiphenylmethane. The first three compounds give only a diazo compound, while the last two yield phenol and diazo compound in varying proportions according to the experimental conditions. Of these compounds some, *e.g.*, the diazo compound of diphenylmethane dicarbonic acid, fuse at a temperature above 300°, while others, for instance diazodiacetyldiphenylmethane, decompose on heating.

Products of Condensation of Chlorides of *o*- and *p*-Nitrated Benzile with Acetylacetone.—M. Mech.—*p*-Nitrated benzile chloride gives with acetylacetone the disubstituted derivative—

$$CH_3COC\begin{matrix} \diagup CH_2^{(1)}C_6H_4^{(4)}NO_2 \\ —COCH_3 \\ \diagdown CH_2^{(1)}C_6H_4^{(4)}NO_2 \end{matrix},$$

and also *p*-nitrophenylbutanone—

$$CH_3COCH_2CH_2^{(1)}C_6H_4^{(4)}NO_2,$$

of which the semicarbazone has been prepared. The corresponding compound from ortho-nitrated benzyle, *o* nitrophenylbutanone, has not been isolated, but its oxime and semi-carbazone have been prepared, and evidently the ortho compound reacts in exactly the same way as the para compound with acetylacetone.

COUNTY COUNCIL OF DURHAM.

DARLINGTON LOCAL COMMITTEE—TECHNICAL COLLEGE.

Wanted, an ASSISTANT LECTURER qualified to teach Chemistry, Metallurgy, Mineralogy (including Iron and Steel Manufacture), Geology, and allied subjects. Form of application, scale of salaries, and particulars of duties from the undersigned; the former to be returned not later than AUGUST 21st, 1908.

A. C. BOYDE,
Local Secretary.

Education Office,
North Lodge, Darlington,
July 24th, 1908.

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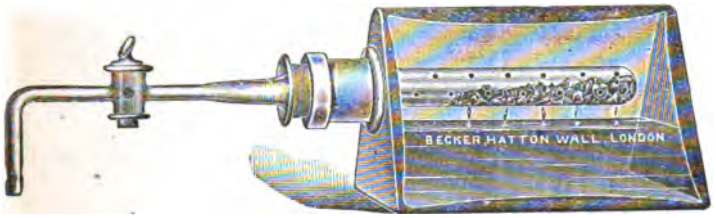

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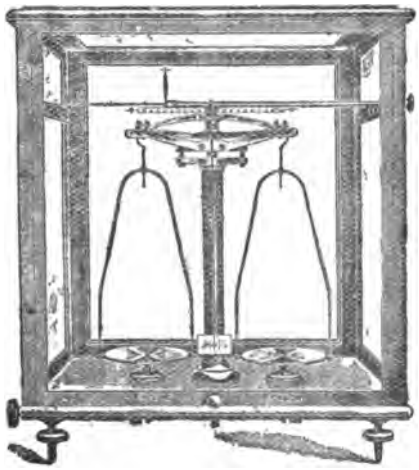

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THE MECHANISM OF MOTION.

By JOHN McDOWALL, F.C.S.

For some time I have been engaged on the study of some of the problems of motion, and the conclusions which I have formed may be of some interest to your readers.

The main difficulty I have experienced is to form a mechanical conception of motion.

Suppose, for instance, we apply motion to a projectile so that it moves for a distance through space, we suppose it to be charged with an occult principle called energy, on account of which it is enabled to diminish or increase the space between itself and other objects.

Now this operation, like all other phenomena in Nature, must have a mechanical foundation, and must work according to laws which can be understood, and in the foregoing example I confess that it is impossible to form a mechanical conception of how a body charged with any occult principle can move through space faster than other bodies which have not received the same initial quantity of energy.

Now, I have been able by looking at the matter from a completely different point of view to form a mechanical conception of motion, and in addition the inferences founded on this view explain in a very plausible way some of the difficulties of physicists.

Suppose the fundamental unit in the atom to be a species of wave which moves at a speed proportional to its propagation. Matter would then be fundamentally a state of the ether which was handed on—nothing would move through the ether in the sense of a body moving through a liquid, but a state would move through it.

If we look at the matter from this point of view we can form a mechanical conception of how motion takes place.

The ether acts as a continuous medium on account of which the state which we have assumed to be matter can move mechanically along, and this without resistance no matter how dense the ether may be.

It might be a thousand times more dense than steel, and still matter would be able to flit like a shadow through it without resistance.

The reader will probably have some difficulty in detaching himself from the erroneous impressions of the senses and look at the matter from a purely abstract point of view.

We are accustomed to see the superficial working of motion from our childhood, and we see a projectile moving through the air without any mechanical bases for its motion, and conclude instinctively that as the projectile moves through the air in like manner the fundamental unit of matter moves through the ether: familiarity has destroyed the power of looking at the matter from the point of view of pure reason.

Reasoning from this starting point we regard the atom from a different point of view. It may be that the atom is composed of corpuscles, and it may be that these corpuscles are in their turn composed of others, but the fundamental unit, however it may be combined, is a species of wave which moves at a speed proportional to its compression, as it is as inconceivable that motion can take place without a mechanical basis as the old idea of action at a distance, and is a fallacy of the senses.

When we make a body move, the motion is transmitted to it in the form of a large wave in the matter which connects the body with the source of the impulse, and this large wave distributes itself over all those little fundamental waves which constitute matter, reinforcing them, and the body as a whole on account of the greater compression of the fundamental waves moves on with an increased speed, and the difficulty in overcoming the elasticity of the ether in altering the state of compression of the fundamental unit produces an initial difficulty in starting the body, which is known as inertia.

There are some difficulties which at first sight are not easy to explain.

It is not easy to conceive how such an elusive thing as a wave can combine to form a rigid body. In order to explain this we have to look on the fundamental unit as a centre of distortion in the ether—the ether surrounding it must be in a state of strain, as a part of it is under compression or distortion of some kind, and it is to this strain that I attribute the rigidity of matter; the fundamental units tend to settle down in the vicinity of each other in a position determined by the strain in the ether which surrounds them.

Again, rigidity is a relative term; we have no absolute measurement for rigidity, we simply compare the rigidity of one kind of matter with another. In those circumstances the forces holding matter together compared with such things as the rigidity of the ether might be of the feeblest description, like bubbles held together by surface tension on the surface of a pond; what would be a gentle shake compared with those other forces would completely vapourise matter.

Sir Oliver Lodge, in his calculations on the density of the ether, has shown in a striking manner the order of magnitude of the weight of the ether, and the energy which it contains is probably of the same order, which is much vaster than what can be displayed by matter.

The ether, in fact, has unique properties of its own, and we cannot look upon it as obeying the same laws as matter, though it is possible to deduce its properties by reasoning.

The results of my investigation may be summed up in this, "That motion can only be transmitted through the agency of a conducting medium;" so if in any part of space there was a break or hole in the ether it would be impossible for matter or any form of motion to pass through it, just as it would be impossible for sound to pass through a vacuum.

Sebaieh, Upper Egypt.

THE DECOMPOSITION OF CERTAIN MINERALS AND INDUSTRIAL PRODUCTS BY MEANS OF SODIUM PEROXIDE AND METALLIC SULPHIDES.*

By JAMES H. WALTON, Jun., and HERMAN A. SCHOLZ.

In the year 1893 J. Clark (*Journ. Chem. Soc.*, 1893, liii., 1079—1083) published a method for decomposing pyrites, zinc blende, galena, chromium, and arsenic ores by mixing them with sodium peroxide in an open platinum or nickel dish and fusing in the usual manner. W. Hempel (*Zeit. Anorg. Chem.*, 1893, iii., 193) used a similar method for the decomposition of chromium, manganese, tungsten, and titanium ores. Spüller and Kalman (*Chemiker Zeitung*, 1892, xvii., 881, 1207, 1360, 1412; 1893, xviii., 292), Edinger (*Zeit. Anal. Chem.*, 1895, xxxiv., 362), Saniter (*Chemiker Zeitung*, 1898, xix., 1657), Glaser (*Journ. Am. Chem. Soc.*, 1898, xx., 130), Angenot (*Zeit. Angew. Chem.*, 1904, xvii., 1274), and Lucchese (*Ann. Chim. Anal. Appl.*, 1905, ix., 450) used sodium peroxide for the decomposition of various organic and inorganic substances. These investigators all followed the method of placing the substance to be decomposed in a crucible, mixing with sodium peroxide, and applying heat to the crucible. While the decompositions made in this way are very complete, the sodium peroxide attacks the crucible

* *American Chemical Journal*, xxxix., No. 7.

vigorously, and a large amount of foreign matter is consequently introduced into the determination.

A number of investigators have published methods of decomposition in which the heat necessary to bring about the decomposition is furnished by the reaction between the peroxide and some organic substance present in the mixture. S. W. Parr (*Journ. Am. Chem. Soc.*, 1900, xxii., 646) decomposed coal by mixing it in a steel bomb with sodium peroxide and igniting the mixture by dropping a red-hot iron wire into the mass, the combustion taking place without further application of heat. Although this method was used primarily for the determination of the heating power of coals, it was found that the sulphur was completely oxidised to the sulphate and could be determined in the usual manner.

Pringsheim (*Am. Chem. Journ.*, 1903, xxxi., 386; *Ber.*, 1904, xxxvii., 2155; 1906, xxxviii., 2436) used a similar method for the quantitative and qualitative determination of the halogens, arsenic, and phosphorus in organic compounds. Von Konek (*Zeit. Angew. Chem.*, 1904, xvii., 771, 1093) also used this method for making qualitative analyses of organic substances, using a cylindrical nickel or steel crucible similar to that designed by Parr for use in his calorimeter. The finely powdered sample, in this case, was mixed with sodium peroxide and a compound rich in carbon, such as camphor, and the mass ignited by dropping in a red-hot iron wire.

The decomposition of substances by means of the heat obtained from the action of the sodium peroxide on an organic compound mixed with the substance has several advantages. In the first place, the vessel in which the decomposition is carried out is attacked to an extent which is for many purposes inappreciable. Moreover, the decomposition is completed in a very few minutes. It seemed to the authors that this method could be modified and used to decompose certain substances of technical importance, particularly those which require the use of nitric acid for their decomposition, the excess of which must often be removed by evaporation with sulphuric acid before the determination can be made.

Experimental.

The sodium peroxide used in these experiments was the ordinary commercial article manufactured by Koenig and Co. It contained traces of SiO_2, Al_2O_3, and Fe_2O_3. Its purity was determined by measuring the volume of oxygen which was given off from a weighed amount of the substance when decomposed by water containing a few drops of a dilute solution of cobalt nitrate (Archbutt, *Analyst*, 1895, xx., 3).

The sample used in this work contained between 95 and 96 per cent sodium peroxide.

The Decomposition of Substances containing Lead.

a. Galena Ores.—Two galena ores were used in testing this method of decomposition. The percentage of lead present was first determined gravimetrically by boiling the finely ground ore (passed through a 200-mesh sieve) with concentrated nitric acid and a small amount of hydrochloric acid for one and a-half hours, precipitating the lead as the sulphate, filtering, separating the lead sulphate from the residue by dissolving in hot ammonium acetate solution, re-precipitating by means of sulphuric acid, and weighing the lead sulphate on a Gooch crucible.

The following values were obtained:—

Per Cent of Lead.

	1.	2.
Ore No. 5	61·58	61·52
Ore No. 22	56·05	55·98

These iron ores contained a small amount of iron pyrites and a little silica. Long boiling with acid was found to be absolutely necessary to completely decompose them. In one case in which the ore was boiled for only one-half hour, results were obtained which were several per cent lower than those given above. The above results were taken as the true values for these ores, as they agree with analyses made later by different methods.

For the purpose of determining the right kind of sodium peroxide mixture to decompose these ores, a number of qualitative experiments were made in which the ores were placed in the bomb of a Parr calorimeter and mixed with sodium peroxide and various easily oxidisable substances, such as magnesium, ammonium oxalate, and starch. The thoroughly mixed charge was ignited by dropping a red-hot piece of iron wire into the water-cooled bomb. When starch was used the mass fused quietly. It was found that the addition of a small amount of potassium persulphate to the sodium peroxide gave a better fusion and an apparently complete decomposition of the ore (S. W. Parr, *Journ. Am. Chem. Soc.*, 1902, xxiv., 167).

The following quantitative experiment was carried out:—

Experiment I.

0·3 grm. ore No. 5 (200-mesh sieve); 5·0 grms. sodium peroxide; 0·2 grm. starch; 0·3 grm. potassium persulphate.

This mixture was ignited in a closed bomb by means of a fine iron wire which passed through the charge, and which was heated to a red heat by means of an electric current. The melt was dissolved in 100 cc. of water and enough sulphuric acid added to neutralise the alkali and leave an excess of 2 per cent acid. Sodium bisulphite was added to reduce the lead peroxide, and the solution was boiled for five minutes. It was then diluted and the lead determined gravimetrically as the sulphate.

The following results were obtained:—

Per Cent of Lead.

Ore No. 5 ..	57·49	60·22	59·13

In these experiments it was observed that the fusion did not take place quietly, but was attended by slight explosions, which threw the fused mass upon the side of the bomb. It seemed reasonable to believe that this fact might be the cause of the low results.

Zinc sulphide, when mixed with sodium peroxide and ignited, is oxidised with the evolution of great heat, the oxidation taking place quietly however. The following quantitative experiment was made in which this substance was substituted for the starch. The zinc sulphide used was the dry, finely divided product prepared by Kahlbaum.

Experiment II.

0·5 grm. ore; 8·0 grms. sodium peroxide; 0·8 grm. zinc sulphide; 0·3 grm. potassium persulphate.

The mixture was placed in an open bomb and ignited as before. The entire mass became liquid and red-hot, and the fusion took place without any spattering. The following results were obtained:—

Per Cent of Lead.

	1.	2.
Ore No. 5	60·61	59·73
Ore No. 22	55·51	55·18

In this series the lead sulphate was determined by titrating the ammonium acetate solution of lead sulphate with ammonium molybdate according to Alexander's method (*Eng. and Min. Journ.*, xiii., 55; see also *Bull. School of Mines Quarterly*, 1902, xxiii., 348), the melted mass from the fusion being treated in other respects as in Experiment I. The results obtained in the analysis of the two ores are somewhat better than those obtained when starch was used, but they are from 0·5 to 1·00 per cent lower than the values obtained by the acid decomposition method.

In several cases in which the above charge was used the decomposition was found to be incomplete, a slight amount of the ore remaining undecomposed in the sharp edge of the bomb at the point at which the wall and bottom intersect. It was apparent that this part of the bomb did not become sufficiently hot to decompose the

ore. Since the fusion takes place quietly, it was found possible to avoid this uncertainty of decomposition by using a nickel crucible which has rounded edges, and consequently affords no place for the ore to lodge. Moreover, the melt could be more easily removed and less water was needed for its solution. The nickel crucible used had a capacity of 30 cc. and was 1¼ inches in diameter. The charge was placed into the crucible and ignited by means of a short piece of magnesium ribbon instead of iron wire, which caused considerable trouble by forming the magnetic oxide of iron. This compound, being difficultly soluble, remains as a residue and makes it very difficult to tell whether or not the ore is completely decomposed.

Experiment III.

The charge used was the same as in Experiment II. The dry, finely ground sodium peroxide was measured by means of a small aluminium scoop. The other substances were added and thoroughly mixed by means of a glass rod, which was then brushed clean. The cover was placed upon the crucible and was moved far enough to one side to permit the dropping in of a piece of lighted magnesium ribbon about one-half inch in length.

After the ignition had taken place the crucible was immediately covered. The lead was determined by the molybdate method, the following results being obtained:—

Per Cent of Lead.

	1.	2.
Ore No. 5	61·23	61·22
Ore No. 5 (gravimetrically).	60·68	59·07
Ore No. 22	55·82	55·68

These low results might be due to incomplete decomposition, although, from the fact that no residue could be seen on dissolving the fusion in acid, this seemed improbable. To test this point, however, a sample of the ore was fused with sodium peroxide in an iron crucible, the heat being supplied by a Bunsen burner.

Experiment IV.

0·3 grm. ore No. 5; 5·0 grms. sodium peroxide.

This mass was kept in a molten condition for five minutes and the lead determined gravimetrically. The results were low, 60·07 and 60·06 per cent of lead being found in duplicate determinations.

It will be seen that the results obtained with ore No. 5, in Experiments II., III., and IV., vary between 59·07 and 61·23. It was noticed that in the experiments in which the lowest results were obtained, the precipitate of lead sulphate was coloured dark brown, due to basic sulphates of iron which are precipitated upon boiling in a slightly acid solution when large quantities of alkali salts are present (Pickering, *Journ. Chem. Soc.*, 1880, xxxvii., 807). It occurred to the authors that these basic iron salts kept the lead sulphate from being dissolved by the ammonium acetate solution, the effect being similar to that produced by the presence of barium sulphate (*Bull. School of Mines Quarterly*, 1902, xxiii., 348).

To test this idea the following experiments were carried out:—

Experiment V.

A fusion of ore No. 5 was made as in Experiment III. and the melt treated as before, except that the boiling on acidifying was omitted in order to prevent, as much as possible, the formation of the basic iron salts. The solution was heated to the boiling-point and then allowed to cool. The residue still had a brown colour, but it was much lighter than before. The lead was determined by the molybdate method, the percentages found being 61·23 and 61·16.

Experiment VI.

This fusion was also carried out as in Experiment III. The crucible and melt were put into a casserole, and about 75 cc. of water containing 7 cc. of sulphuric acid (sp. gr. 1·84) were added. Enough sulphuric acid was present to neutralise all of the alkali. The melt dissolved in two or three minutes, and the crucible was immediately removed. An excess of 4 cc. of sulphuric acid (sp. gr. 1·40) was then added, and a concentrated solution of sodium bisulphite poured in to reduce the lead peroxide. The solution was boiled for a minute, diluted to 200 cc., cooled, and 20 cc. of 95 per cent alcohol added. The residue was almost pure white. The lead was determined by the molybdate method, 61·46 per cent being found present. On repeating the determination, following the procedure described, the values 61·51 and 61·57 per cent were obtained.

Ore No. 22 was analysed by the same method, 55·89 and 55·88 per cent of lead being obtained. These results agree closely with those obtained by the acid decomposition method. It seems logical to conclude that the low results were due to the presence of the basic salts of iron. The formation of these basic salts may be avoided by dissolving the fusion in acidified water.

In order to determine the value of the above method of decomposition as a general method for the decomposition of other substances containing lead, experiments were carried out in which some typical substances were analysed by the sodium peroxide method, and also by the standard method.

Experiment VII.

A low grade galena ore (No. 45), high in both silica and iron pyrites, was decomposed by boiling with nitric and hydrochloric acids, and the lead determined gravimetrically, 3·13 per cent lead being found. The ore was also decomposed by means of the following charge:—

1·00 grm. ore No. 45 (through 200-mesh sieve); 10 grms. sodium peroxide; 0·8 grm. zinc sulphide; 0·3 grm. potassium persulphate.

The mixture was ignited by means of a magnesium ribbon. A very hot fusion was obtained, due to the oxidation of the pyrite in the sample. The melt was treated as in Experiment VI. and the lead determined by titration with ammonium molybdate solution, the values 3·16 and 3·19 per cent of lead being obtained in duplicate determinations.

b. Lead Glaze.—A lead glaze high in calcium and silica was analysed by the two methods. A 1 grm. sample of the glaze (200-mesh sieve) was decomposed by the nitric acid method and the lead determined by the molybdate method. The large amount of silica caused difficulties both in filtering the insoluble residue and in dissolving the lead sulphate in ammonium acetate solution, necessitating a much longer boiling.

	1.	2.
Per cent of lead in glaze ..	27·81	27·82

Experiment VIII.

1·0 grm. glaze (200-mesh sieve); 10·0 grms. sodium peroxide; 1·5 grms. zinc sulphide; 0·4 grm. potassium persulphate.

Since there was no combustible matter in the sample, it was necessary to raise the proportion of zinc sulphide. The fusion was good and the glaze completely decomposed. The melt dissolved in water, the absence of iron making unnecessary the precautions taken in the previous analyses to avoid the precipitation of basic salts. The lead determined by the molybdate method gave 27·84 and 27·82 per cent lead. In this fusion the silica went into solution almost completely and was filtered off from the insoluble residue, and thus did not interfere in the filtering nor in the solution of the lead sulphate.

c. Lead Slag.—A sample of 0·5 grm. of lead slag (100-mesh sieve) was decomposed by nitric and hydrochloric acids and the lead determined by the molybdate method, 36·39 per cent being found.

Experiment IX.

0·5 grm. lead slag (100-mesh sieve); 8·0 grms. sodium peroxide; 1·2 grms. zinc sulphide; 0·3 grm. potassium persulphate.

Ignited with magnesium wire. The fusion was good and the decomposition appeared to be complete. Since there was very little iron present, the melt was dissolved in water and treated in the usual manner and the lead determined by the molybdate method. Results were 36·58 and 36·48 per cent lead.

These results by the sodium peroxide method compare very favourably with those obtained by the standard nitric and hydrochloric acid decompositions, as may be better seen from Table I.

TABLE I.—*Comparison of Lead Determinations by Standard and by Sodium Peroxide Methods of Decomposition.*

Sample.	Standard method, per cent Pb.	Peroxide method, per cent Pb.
Galena ore No. 5 ..	61·58(*a*)	61·46
„ „ 5 ..	61·62(*a*)	61·51
„ „ 5 ..	—	61·57
„ „ 22 ..	56·04(*a*)	55·89
„ „ 22 ..	55·98(*a*)	55·90
„ „ 22 ..	—	55·88
„ „ 45 ..	3·13(*a*)	3·16
„ „ 45 ..	—	3·19
Lead glaze	27·81	27·84
„	27·82	27·88
Lead slag..	36·39	36·48
„	—	36·58

(*a*) Lead determined gravimetrically as $PbSO_4$.

(To be continued).

A REVISION OF THE ATOMIC WEIGHT OF LEAD.*

PRELIMINARY PAPER.—THE ANALYSIS OF LEAD CHLORIDE.

By GREGORY PAUL BAXTER and JOHN HUNT WILSON.

ALTHOUGH lead is one of the most common elements, its atomic weight has received comparatively little attention, the value at present accepted being based almost wholly upon the work of Stas (see Note).

NOTE.—Earlier work on the atomic weight of lead has been carefully summarised by Clarke, Smithsonian Miscellaneous Collections—Constants of Nature, "A Re-calculation of the Atomic Weights," 1897. In re-calculating the data of earlier determinations the following atomic weights have been used in this paper :—O = 16·000, Ag = 107·88, Cl = 35·46, N = 14·01, S = 32·07. Richards and Wells (*Pub. Car. Inst.*, 1905, No. 28), Richards and Forbes (*Ibid.*, 1907, No. 69, p. 47), Richards and Jones (*Ibid.*, No. 69, p. 69; "Report of International Committee on Atomic Weights," *Journ. Am. Chem. Soc.*, 1907, xxix., 110).

Of the earlier determinations of this constant those of Döbereiner (*Schweig. Journ.*, 1816, xvii., 241) and Longchamps (*Ann. Chim. Phys.*, 1827, xxxiv., 105) can hardly be considered as possessing other than historic interest. The first results which can lay claim to accuracy are those of Berzelius (*Pogg. Ann.*, 1830, xix., 314), who obtained values ranging from 206·7 to 207·3 by reduction of litharge in a current of hydrogen. Berzelius also synthesised the sulphate from metallic lead with the result 207·0 ("Lehrbuch.," Fifth Ed., 1845, iii., 1187). Shortly after, Turner (*Phil. Trans.*, 1833, 527) criticised the first method employed by Berzelius, and attributed the irregularity of his results to the action of lead oxide on the siliceous matter of the tube at the temperature employed in the reduction. By the conversion of both the metal and the oxide into sulphate Turner, in a painstaking research, deduced the values 207·0 and 207·6 respectively, and by converting the nitrate into sulphate, 204·2. Marignac converted metallic lead into the chloride by heating in a stream of chlorine and obtained the result 207·42 (*Ann.*, 1846, lix., 289). Both Marignac (*Journ. Prakt. Chem.*, 1858, lxxiv., 218) and Dumas (*Ann.*, 1860, cxiii., 35) analysed lead chloride. Marignac, who dried the salt at 200°, by titration against silver, found the atomic weight of lead to be 206·81, and from the ratio of lead chloride to silver chloride, 206·85. Dumas subsequently showed that lead chloride, even when dried at 250°, retains moisture and is somewhat basic, and in one analysis in which corrections are applied for these errors, found a somewhat higher value, 207·07, as was to be expected. Chloride analyses by early investigators are, however, to be universally distrusted, owing to neglect of the very considerable solubility of silver chloride, thus producing too low results.

Stas's work upon the syntheses of lead nitrate and sulphate from the metal is undoubtedly the most accurate contribution upon the subject, although a careful consideration of his work discloses minor defects, many of which he recognised himself ("Œuvres Completes," i., 383). The metallic lead used in the syntheses was finally fused under potassium cyanide. Whether or not this treatment introduced impurities into the metal is uncertain. Stas himself suspected the presence of alkalis in the metal. Since the nitrate could not be dried above 150° without decomposition, it undoubtedly contained moisture, and Stas calls attention to this point. The sulphate was made by treatment of lead nitrate, resulting from the nitrate syntheses, with sulphuric acid. The sulphate was dried finally at dull redness, and was probably free, or nearly free, from moisture, although it may have contained traces of lead oxide resulting from occluded nitrate, as well as sulphuric acid. Most of these probable errors tend to lower the observed atomic weight, so that Stas's value from the series of nitrate syntheses, 206·81, and that from the sulphate series, 206·92, are to be regarded as minimum values. The reader of Stas's own account of his work upon lead cannot fail to be impressed with the fact that he was somewhat dissatisfied with the outcome of his research. Mention should also be made of the work of Anderson and Svanberg (*Ann. Chim. Phys.*, 1843, [3], ix., 254) on the conversion of lead nitrate into oxide, although the method was primarily employed in an endeavour to fix the atomic weight of nitrogen. Their results yield the value 207·37.

The discrepancies between the results of these various experiments only serve to emphasise the need of a re-determination of the value in question, and it was with this object in view that the work embodied in this paper was undertaken.

The search for a suitable method for determining the atomic weight of lead failed to reveal any more promising line of attack than those already employed for the purpose. With an element of so high an atomic weight as lead, in any method involving the change of one of its compounds into another, errors which may be insignificant with elements of small atomic weight are magnified in the calculations to undesirable proportions. Furthermore, during the following investigation, reduction of the chloride and oxide in hydrogen was investigated far enough to show that complete reduction of either compound was extremely difficult, if not impossible, without loss of material from the containing vessel by sublimation, aside from the fact that all available material for containing vessels is acted upon by either the fused salt or the reduced metal. The elimination of moisture from lead nitrate or lead sulphate without decomposition of the salts seemed likely to prove a stumbling block in the use of these substances. Finally, in spite of the slight solubility of lead chloride, the determination of the chlorine in this salt by precipitation with silver nitrate was chosen as presenting fewer difficulties. In the first place, the determination of a halogen can be effected with great accuracy. In the second place, the elimination of moisture from lead chloride is an easy matter, since the salt may be fused in a platinum vessel in a current of hydrochloric acid gas without attacking the platinum in the least, and without the production of basic salts. In the third place, silver chloride, which has been

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precipitated from a dilute solution of lead chloride by means of silver nitrate, does not contain an amount of occluded lead salt large enough to be detected.

Purification of Materials.

Water.—All of the water used in either the purification or the analyses was distilled twice, once from an alkaline permanganate solution and once from very dilute sulphuric acid. Block-tin condensers were used in both distillations, and rubber and cork connections were avoided. Generally, receivers of Jena glass were employed, but in certain cases the water was collected in platinum or quartz vessels.

Hydrochloric Acid.—Commercial C. P. hydrochloric acid was diluted with an equal volume of water and distilled with a quartz condenser, only the middle fraction being collected.

Nitric Acid.—Nitric acid was distilled with a platinum condenser, until free from chlorine. Two distillations were invariably sufficient to accomplish this end, if the first third of each distillate was rejected.

Silver.—Pure silver was obtained by methods already many times employed in the laboratory of Cambridge, Mass. Silver nitrate was dissolved in a large volume of water, and the silver was precipitated as chloride with an excess of hydrochloric acid. The precipitate was thoroughly washed and reduced with alkaline invert sugar. The reduced silver, after being washed, was dried and fused on charcoal in the flame of a clean blast-lamp. After the buttons had been cleaned by scrubbing with sand and etching with nitric acid, they were dissolved in pure dilute nitric acid, and the silver was precipitated as metal with ammonium formate (Richards and Wells, *Pub. Car. Inst.*, 1905, No. 28, 19). This silver was washed and fused in the flame of a blast-lamp on a crucible of the purest lime. The buttons were cleaned as before, and then electrolysed (Abrahall, *Journ. Chem. Soc. Proc.*, 1892, p. 660). Finally, the electrolytic crystals were fused in a boat of the purest lime in a porcelain tube in a current of pure electrolytic hydrogen (Baxter, *Proc. Amer. Acad.*, 1903, xxxix., 249). The bars of silver were cut in pieces with a fine steel saw, etched with dilute nitric acid until free from iron, washed, dried, and heated in a vacuum to 400°. The silver was kept in a desiccator containing solid potassium hydroxide.

Lead Chloride.—Three samples of lead chloride from two entirely different sources were employed. Sample A was prepared from metallic lead. Commercial lead was dissolved in dilute nitric acid, and the solution, after filtration, was precipitated with a slight excess of sulphuric acid. The lead sulphate was thoroughly washed, suspended in water, and hydrogen sulphide was passed in until the sulphate was almost completely converted into sulphide. Next, the sulphide was washed with water, dissolved in hot dilute nitric acid, and the solution was freed from sulphur and unchanged sulphate by filtration. The lead nitrate thus obtained was crystallised twice, dissolved in water, and precipitated in glass vessels with a slight excess of hydrochloric acid. The chloride was washed several times with cold water, and then crystallised from aqueous solution eight times, the last five crystallisations being carried out wholly in platinum, with centrifugal drainage after each crystallisation. In crystallising the lead chloride the whole sample was not dissolved at one time, but the same mother-liquor was used for dissolving several portions of the original salt. Needless to say, the chloride was not exposed to contact with the products of combustion of illuminating gas, lest lead sulphate be formed.

Sample B was prepared from commercial lead nitrate. This salt was dissolved and crystallised from dilute nitric acid, once in glass and six times in platinum vessels, with centrifugal drainage. Hydrochloric acid was then distilled into a large quartz dish, and the solution of the nitrate was slowly added with constant stirring with a quartz rod. The chloride was freed from aqua regia as far as possible by washing with cold water, and was once crystallised from aqueous solution in quartz dishes to remove last traces of aqua regia. Finally, the salt was crystallised three times in platinum.

It could reasonably be expected that both of these samples were of a high degree of purity; nevertheless, upon heating the salt in an atmosphere of hydrochloric acid, the salt itself turned somewhat dark, and upon solution of the fused salt in water a slight dark residue remained. Although in a few preliminary experiments attempts were made to determine this residue by filtration and ignition, it was subsequently found that even a small filter-paper adsorbs appreciable amounts of lead compounds from a solution of the chloride, which cannot be removed by washing with water. From three to thirteen hundredths of a mgrm. of residue were obtained in several blank experiments, by ignition of filters through which ½ per cent solutions of lead chloride had been passed with subsequent very thorough washing. In order to avoid the uncertainty of this correction, further attempts were made to obtain a sample of the salt which would give a perfectly clear solution in water after fusion, and thus render filtration unnecessary. With this end in view a considerable quantity of Sample A was fused in a large platinum boat in a current of hydrochloric acid. The fused salt was powdered in an agate mortar, dissolved in water in a platinum vessel, and the solution was freed from the residue by filtration through a tiny filter in a platinum funnel into a platinum dish, where it was allowed to crystallise. This sample was then twice re-crystallised with centrifugal drainage. Notwithstanding the drastic treatment to which it had been subjected, when a portion of this material was fused in hydrochloric acid, the same darkening as before was observed, and the same residue was obtained. The suspicion that the difficulty was due to dissolving of the filter-paper by the solution of the salt led to a second more successful attempt by crystallisation from hydrochloric acid solution in platinum vessels. (Mr. P. B. Goode, in the laboratory of Cambridge, Mass., has recently found a similar difficulty with the chlorides of the alkaline earths). In this way it was found possible to prepare salt which showed no tendency to darken upon heating, and which, after fusion, left absolutely no residue upon solution in water. Portions of Samples A and B were thus re-crystallised three times more. Since these two specimens of material gave identical results, for two final experiments, portions from each of these samples were mixed and then subjected to three additional crystallisations. This last sample was designated Sample C.

(To be continued).

ON THE DETECTION OF OZONE, NITROGEN PEROXIDE, AND HYDROGEN PEROXIDE IN GAS MIXTURES.

By EDWARD H. KEISER and LEROY McMASTER.

When air is acted upon in a number of ways, such as by electric sparks, the flaming arc, burning hydrogen, burning magnesium, heated platinum wire, &c., in short, by any means which produce very high temperatures, ozone, nitrogen peroxide, and hydrogen peroxide may be formed. The silent electric discharge and the slow oxidation of phosphorus produce oxidising gases in air. Several commercial processes of treating air so as to convert it into a more active chemical agent are at present in use. It is desirable, therefore, to have characteristic reactions for each of the three substances above mentioned in examining the gases produced in these different ways.

A great many reagents have been suggested for this purpose, but many of these have been found to be unreliable, while others are not very sensitive, and still others are rare substances that are not easily obtained. The accompanying table contains a list of such substances, together with a statement of their behaviour toward ozone, nitrogen peroxide, and hydrogen peroxide.

	Ozone.	Nitrogen peroxide.	Hydrogen peroxide.
Potassium iodide and starch (Schönbein, *Ber. u. d. Verh. d. Nat. Ges. Basel*, iv., 58)	Blue.	Blue.	Blue.
Wine-red litmus-paper moistened with potassium iodide (Houzeau, *Ann. Chim. Phys.*, [4], v., 20)	Blue.	No change.	Blue.
Tetramethylparaphenylenediamine (Wurster, *Ber.*, xix., 3195)	Bluish violet.	Bluish violet.	Bluish violet.
Metaphenylenediamine, alkaline solution (Erlwein and Weil, *Ber.*, xxxi., 3158)	Burgundy red or yellow.	No change.	No change.
Manganese chloride paper moistened with guiacum tincture (Engler and Wild, *Ber.*, xxix., 1940)	Blue.	Blue.	No change.
Benzidine in alcohol (Arnold and Mentzel, *Ber.*, xxxv., 1324)	Brown.	Blue.	No change.
Tetramethyl-*pp*-diaminodiphenylmethane in saturated alcoholic solution (Arnold and Mentzel, *Ber.*, xxxv., 1324)	Violet.	Yellow.	No change.
Potassium ferricyanide and ferric chloride (Schönbein, Weltzien, also Schöne, *Ber.*, vii., 1695; Engler and Wild, *Ber.*, xxix., 1940)	No change.	No change.	Blue.
Manganese dioxide or copper oxide (Andrews and Tait, *Liebig's Ann. Chem.*, cxii., 135)	Decomposed.	No change.	Decomposed.
Silver foil (*Ber.*, xxxv., 1326)	Black.	No change.	No change.
Chromic acid and ether (Barreswill, *Ann. Chim. Phys.*, [3], xx., 364)	No change.	No change.	Blue.
Chromic acid (Engler and Wild, *Ber.*, xxix., 1940)	No change.	—	Decomposed.
Titanium hydroxide in sulphuric acid (Schönn, *Zeit. Anal. Chem.*, ix., 41)	No change.	No change.	Yellow.
Thallous salts (Schöne, *Liebig's Ann. Chem.*, cxcvi., 58)	Brown.	No change.	No change.
Ammonium molybdate in sulphuric acid (Schönn, *Zeit. Anal. Chem.*, ix., 41)	No change.	No change.	Yellow.
Guiacum tincture with malt infusion (Struve, *Zeit. Anal. Chem.*, viii., 274)	No change.	No change.	Blue.
Gold chloride free from acid (Böttger, *Zeit. Anal. Chem.*, xix., 105)	Black.	No change.	—
Nitrite test with sulphanilic acid and α-naphthylamine (*Bull. Soc. Chim.*, 1889, [3], ii., 347)	No change.	Pink.	No change.

Arnold and Mentzel have studied a number of the reagents that are mentioned in the preceding list (*Ber.*, xxxv., 1324). They find that potassium iodide and starch, and zinc iodide and starch, as well as guiacum tincture, give a blue colour with ozone, nitrogen peroxide, chlorine, and bromine. In short, these reagents are not characteristic of anything more than an oxidising gas. Red litmus-paper moistened with potassium iodide solution they find to be entirely unreliable, because all paper turns blue with free iodine, and any gas that liberates iodine will turn the paper blue. They also found that a solution of potassium iodide to which wine-red litmus solution had been added was unsatisfactory, because the ozone acts upon the litmus and changes it to a green colour. The third reagent in the above list, tetramethylparaphenylenediamine, is also turned blue by all oxidising gases. Metaphenylenediamine, the fourth one, was unsatisfactory because they found that nitrogen peroxide gave the same colour as ozone, and all oxidising gases give yellowish colours with this substance. Silver foil is not satisfactory because it is not at all sensitive. (Manchot and Kampschulte, *Ber.*, xl., 2891, have recently shown that while silver foil is not at all sensitive at ordinary temperatures, it becomes much more so at 220—240° C.; for metallic mercury they found the temperature at which action was strongest to be 170° C.). Arnold and Mentzel recommend an alcoholic solution of benzidine or, still better, an alcoholic solution of tetramethyl-*p*-*p*-diamino-diphenylmethane. This gives a violet colour with ozone and yellow with nitrogen peroxide; with hydrogen peroxide it remains unchanged. They found, however, that the oxone obtained by the action of sulphuric acid upon barium dioxide had a different action upon this reagent; instead of violet a green colour was obtained. Benzidine, under the same conditions, gave a blue instead of a brown colour. Similar effects were obtained with the ozone from persulphates, percarbonates, and from sodium and hydrogen peroxides.

F. Fischer and Marx (*Ber.*, xxxix., 2555) have used tetramethyl-*p*-*p*-diaminodiphenylmethane or "tetramethyl base," as it is called, in their investigations upon the formation of ozone and nitrogen peroxide in air at high temperatures. They prepared ozone by the silent electric discharge in the Siemens ozonometer, by the action of ultra-violet light, by the electrolysis of sulphuric acid, and in other ways, and found that ozone produces with this reagent a violet colour and the oxides of nitrogen a yellow colour. Mixtures of both gases give a dirty brown colour intermediate between violet and yellow. Another fact that they emphasise is that the paper must always be kept moist; if it becomes dry, ozone changes it to a yellow colour. To detect very small quantities of nitrogen oxides in the presence of ozone they recommend that the gas be conducted into liquid air. The ozone will dissolve while the oxides of nitrogen will separate as blue flakes. When the liquid air is then filtered, the frozen oxides of nitrogen remain upon the filter. The filtrate is allowed to boil, the air distils away, and the ozone remains. In this way both can be separately identified.

This method is open to the objection that liquid air is not readily obtained in many places. Likewise, the "tetra base" of Wurster and the "tetramethyl base" of Arnold and Mentzel are not easily obtainable substances. Moreover, the colour changes are not always satisfactory. Thallous salts and titanic acid are not always at hand. We have now, however, devised a method which is free from these objectionable features and enables us to identify each of these three substances in the presence of the others.

We found that potassium permanganate, even in very dilute solution, is not decolorised by ozone. Nitrogen peroxide and hydrogen peroxide, on the other hand, both reduce it instantly. To identify ozone in gases that contain at the same time nitrogen peroxide and hydrogen peroxide it is only necessary to draw the gases through a solution of potassium permanganate; the nitrogen peroxide and the hydrogen dioxide will not pass through, while the ozone will, and can then be detected with potassium iodide and starch.

To detect nitrogen peroxide in the presence of ozone and hydrogen peroxide we take advantage of the fact that both ozone and hydrogen peroxide are decomposed when passed through a tube containing powdered manganese dioxide. Nitrogen peroxide passes through unchanged. Its presence can be shown by passing the gas, after it has

gone over the manganese dioxide, into very dilute permanganate. If the latter is decolorised, nitrogen peroxide was present. A still more delicate test for nitrogen peroxide is this: pass the gas, which may contain ozone and hydrogen dioxide, directly into pure caustic soda, made from metallic sodium and nitrite-free distilled water, and then test the caustic soda solution for nitrites by the well-known sulphanilic acid and α-naphthylamine method.

Hydrogen dioxide can be identified in the presence of both ozone and nitrogen peroxide by passing the gas mixture into a solution of potassium ferricyanide and ferric chloride. The yellow-brown solution becomes green and then blue as more hydrogen dioxide passes in. This formation of Prussian blue is characteristic of hydrogen dioxide and is not produced by either ozone or nitrogen peroxide. This we have found by experiment.

I. *Examination of the Gases produced by Burning Hydrogen in Air.*

We have tried our method in the examination of the gases produced by burning hydrogen in air. A jet of burning hydrogen was introduced into the end of a glass tube fifteen millimetres in diameter. A current of air was drawn into the tube by means of an aspirator. The gas passed from the tube through a moderately strong solution of potassium permanganate and then into a solution of potassium iodide and starch. This soon became blue, thus showing that ozone was formed by the hydrogen burning in a rapid current of air. The potassium permanganate wash-bottle was then replaced by a tube forty centimetres in length, filled with powdered manganese dioxide. Between this tube and the aspirator, a wash-bottle with a very weak solution of potassium permanganate was inserted. Air was now rapidly drawn through the apparatus and the products of combustion of the hydrogen passed first over the manganese dioxide and then into the dilute permanganate. The latter, after a time, became decolorised, thus showing that nitrogen peroxide was formed. This was confirmed by passing the gases formed by burning hydrogen into a solution of nitrite-free caustic soda and then applying the nitrite test (Griess test) after having slightly acidified the solution. The pink colour was formed, thus confirming the result obtained with dilute permanganate. The gases formed by burning hydrogen were also tested for hydrogen peroxide by drawing them through a solution of potassium ferricyanide and ferric chloride. This became first green, then blue, thus showing that hydrogen peroxide was present. This ferricyanide and ferric chloride test for hydrogen peroxide we found to be more sensitive than titanium dioxide in sulphuric acid. The latter failed to show the presence of hydrogen peroxide in the gases produced by burning hydrogen in air.

II. *Examination of the Gases produced by the Silent Electric Discharge in Air and Oxygen.*

Atmospheric air was purified and dried by being drawn, by means of an aspirator, through a series of wash-bottles containing silver sulphate, potassium permanganate, and pure concentrated sulphuric acid, and was then passed through a Siemens ozonometer. The ozonometer was connected with the terminals of a Ruhmkorff coil, which, in turn, was connected with a storage battery. The potential of the coil discharge terminals was 2160 volts. The air passing through the ozonometer in which the silent discharge was taking place was thereupon conducted through a very weak solution of potassium permanganate. No decolorisation took place, although the ozonised air was passed for more than two hours. Ozone was shown to be present in quantity, not only by its action upon potassium iodide and starch after it had passed through the permanganate, but also by its odour and by the repeated perforation of the rubber tube that connected the last wash-bottle with the aspirator. We were surprised to find that, under these conditions, no nitrogen peroxide was formed. A quantitative determination of the ozone showed that there was 0·00086 grm. per litre. We also conducted the air directly from the ozonometer into pure caustic soda and tested this for nitrites by the Griess test, but failed to obtain any. With potassium ferricyanide and ferric chloride the ozonised air failed to give a reaction for hydrogen peroxide. The same results, namely, the absence of nitrogen peroxide and hydrogen peroxide in air that had passed through the Siemens ozonometer, were obtained when we omitted washing the air with silver sulphate, caustic soda, potassium permanganate, and sulphuric acid before it entered the ozonometer. These experiments show conclusively that it is possible to ozonise air without, at the same time, forming nitrogen peroxide and hydrogen peroxide. We have repeated the above experiments with oxygen, made from fused sodium peroxide and water, instead of air. We obtained no nitrogen peroxide nor hydrogen peroxide. A quantitative determination showed the presence of 0·0012 grm. of ozone to the litre. This ozonised oxygen failed to decolorise potassium permanganate solution.

III. *Examination of Gases produced by the Action of Concentrated Sulphuric Acid upon Barium Dioxide.*

Concentrated sulphuric acid was allowed to drop upon barium dioxide, contained in an Erlenmeyer flask, and the gases resulting were conducted through a concentrated solution of potassium permanganate in a Geissler potash bulb and then into a solution of potassium iodide and starch. A blue colour soon appeared, thus showing the presence of ozone. The gas, after passing over manganese dioxide and then into permanganate, failed to give the reaction for nitrogen peroxide. Nor were we able to detect hydrogen peroxide by passing the gas through a solution of potassium ferricyanide and ferric chloride.

A small quantity of barium nitrate was now added to the barium dioxide, and, on treating with concentrated sulphuric acid and testing as before, we obtained evidence of the presence of ozone and nitrogen peroxide, but not of hydrogen peroxide, in the gases given off.

IV. *Examination of the Gases produced by the Slow Oxidation of Phosphorus in Moist Air.*

We have also used our method for the examination of the gases formed by the slow oxidation of phosphorus in air in the presence of water. We found ozone and nitrogen peroxide but no hydrogen peroxide.

V. *Examination of the Gases produced by the Action of the Flaming Electric Arc upon Air.*

Air that had passed through the flaming electric arc of an Alsop process machine, such as is used in treating flour, was next examined by our method. We found that this air contained nitrogen peroxide, a trace of ozone, and a little hydrogen peroxide.

VI. *Examination of Atmospheric Air.*

We have applied our method to the examination of country air. Air from outside of the laboratory was drawn through a layer of absorbent cotton, six inches in length, then through a concentrated solution of potassium permanganate in a potash bulb, and finally into potassium iodide and starch. After the air had passed through for five hours, a faint blue colour appeared, thus showing the presence of ozone. Air that was filtered through absorbent cotton and that had passed over a layer of manganese dioxide eighteen inches in length was conducted through a very faintly pink solution of permanganate. No decolorisation was observed, although the air was drawn through for eighteen hours. We conclude from this that there was no nitrogen peroxide in the air. Air filtered through cotton was drawn through a solution of potassium ferricyanide and ferric chloride. After four hours the solution acquired a green colour. We found, however, that the reagent used for detecting hydrogen dioxide, namely, a dilute solution of ferric chloride and potassium ferricyanide, when allowed to stand in air, gradually forms a green precipitate,

even when protected from dust by being covered with a bell-jar. We therefore drew atmospheric air for five hours through pure distilled water, contained in a potash bulb, and then tested this water with the reagent. We obtained no blue precipitate, and therefore conclude that the green colour mentioned above may not have been caused by hydrogen dioxide. These experiments prove that our method is sufficiently sensitive to show the presence of ozone in atmospheric air.—*American Chemical Journal*, xxxix., No. 1.

RESEARCHES ON THE DENSITY OF GASES.*

By PHILIPPE A. GUYE.

(Concluded from p. 55).

II. *Discussion of Results.*

Oxygen.—The system of atomic and molecular weights being referred to oxygen (O = 16), the exact determination of the density of this gas is of considerable importance.

Morley (*Zeit. Phys. Chem.*, 1896, xx., 1) has made 41 determinations of this value, of which the averages of three series are :—

Series.	No. of determinations.	Weight of a standard litre.	Capacity of balloon (*a*).
		Grms.	Litres.
I. ..	9	1·42879	21·6 and 8·8
II. (*a*)..	6	1·42887	20·06 „ 20·56
(*b*)..	9		
III. (*a*)..	7	1·42917	8·83, 16·52 „ 15·38
(*b*)..	10		

(*a*) Capacities are given in round numbers.

In the first series the temperatures varied between 15° and 20°, measured with mercury and air thermometers, the apparatus being connected with a manometer open to the air. In the second, the balloons were submerged in pounded ice, and the pressure measured by a differential manometer. In the third, the balloons were submerged in ice, the pressure being read on a barometer, the cistern of which communicated with the interior of the balloon.

The oxygen was prepared from potassium chlorate and by electrolysis of a solution of caustic potash for Series III.(*b*). The extreme variations between the individual observations were as follows :—

For Series I. .. .. .. $\frac{4·8}{10,000}$

„ II.(*a*) . .. .. $\frac{2}{10,000}$

„ II.(*b*) . .. .. $\frac{6·9}{10,000}$

„ III.(*a*) . .. .. $\frac{6·8}{10,000}$

„ III.(*b*) . .. .. $\frac{7}{10,000}$

Referred to the averages, these extreme variations would be reduced to about one-half.

Morley gives double weight to the average of Series III., and thus arrives at the value, 1 litre oxygen = 1·42900 ± 0·000034.

Rayleigh (*Proc. Roy. Soc.*, 1893, liii., 144) used oxygen prepared by three different methods :—(*a*) By heating a mixture of potassium chlorate and sodium chlorate ; (*b*) by heating potassium permanganate ; (*c*) by electrolysis of water, leading the gas thence through a column of copper oxide heated to redness. The balloon was filled at 0° under pressure of about 1 atm. at London, its capacity being 1836·52 cc. The averages of the results (weight of oxygen contained in the calibrated balloon) for each series are :—

Method.	No. of exments.	Weight of oxygen.	Extreme error.
		Grms.	
Chlorates (*a*) ..	5	2·6269	2·3/10,000
(*b*) ..	5	2·6269	2·3/10,000
Permanganate ..	3	2·6271	1·1/10,000
Electrolysis (*a*) ..	1	2·6271	—
(*b*) ..	2	2·6272	—
Average		2·62704	
Correction for contraction ..		0·00056	
Corrected weight..		2·62760	

One litre of oxygen at Paris = 1·42952.

Dividing the value by 1·00033, the gravity factor, 1 normal litre of oxygen = 1·42905 grms.

Leduc ("Researches sur les Gaz," Paris, 1898), using the balloon method, has reported the density relative to that of air at Paris. He prepared the oxygen (*a*) by electrolysis of an aqueous solution of caustic potash or sulphuric acid, (*b*) by decomposition of potassium permanganate, (*c*) by electrolysis of dilute sulphuric acid, the gas then passing through a column of hot copper oxide.

By the method (*a*) the values, referred to air, varied between 1·10501 and 1·10516, average = 1·1051, extreme variation 1·4/10,000. By the method (*b*), 1·10527 ; by the method (*c*) (3 determinations), 1·10521. (Leduc does not mention the number of determinations ; it would appear that only one was made).

Rejecting the results for (*a*) Leduc adopts 1·10523 for the density of oxygen air referred to at Paris. From this the weight of the normal litre is $\frac{1·29316 \times 1·10523}{1·00033}$ = 1·42876 grms. (taking into account the weight of a litre of air at Paris, determined by the same investigator to be 1·29316, which he claims is accurate to 1/20,000) ; or in round numbers, allowing for the accuracy of 1/20,000 claimed by Leduc, 1 litre of oxygen = 1·4288 grms.

Jaquerod and Pintza (*Comptes Rendus*, 1904, cxxxix., 129), working in this laboratory, have made five determinations of the density of oxygen, obtained by heating potassium permanganate, by filling a volumeter of about 3·5 litres capacity, at 0°, and about 1 atmosphere pressure. These determinations, showing an extreme difference of 6/10,000, give for the weight of the standard litre,—

1 litre oxygen = 1·4292.

These determinations were made by the volumeter method. The authors consider their results to be a little too high, and for their further calculations have adopted the value 1·4290. (D. Berthelot has recently reported all the density determinations made in this laboratory, referred to this unit 1·4292, *Comptes Rendus*, cxliv., 260. In view of the few determinations made, and the reservations made by the authors, this has diminished the accuracy of our other results. In all our other work we have adopted the value 1·4290. See *Journ. Chim. Phys.*, iv., 333).

Gray (*Journ. Chem. Soc.*, 1905, lxxxvii., 1607), in the course of his work on the density of nitric oxide, has made six determinations of the density of oxygen, which show remarkable agreement among themselves. The gas was prepared by heating re-crystallised potassium permanganate. The results give for the weight of a balloon of 267·43 cc. capacity, filled at 0° and 1 atm. pressure at Bonn, the value 0·38228, the extreme variation being only 1·6/10,000. A correction brings the capacity of the balloon to 267·388 cc., so that the weight of a normal litre, taking into account the factor for the gravity at Bonn, is—

1 litre of oxygen = $\frac{0·38228}{0·267388 \times 1·000505}$ = 1·42896 grms.,

a value differing from that of Morley's by only 1/36,000.

* Carried on during 1904, 1905, and 1906 in the Physical Chemistry Laboratory of the University of Geneva. From the *Journal of the American Chemical Society*, xxx., No. 16 ; translated by Helen Isham, from *Archives des Sciences Physiques et Naturelles*, xxiv., 32–62.

Résumé. — Morley's determinations, being the most numerous and carried on under the most varied conditions, would appear to deserve the greatest weight, even though the individual variations are relatively large. Rayleigh's value is 1/20,000 greater than Morley's, while Gray's is 1/36,000 less. On the other hand, the less accurate work of Leduc and Jaquerod and Pintza differ from this by 1/7000, one greater and one less. Whether the mean of all five determinations, or of only the first three, which merit the greater confidence because of the greater number of single determinations and the close agreement between the single determinations, be taken, the value is the same—1·42900. The variations +0·00005 (Rayleigh) and −0·00004 (Gray), from the mean of Morley's values are of the same order as the probable error calculated for the latter (+0·000034).

The value, 1 litre of oxygen = 1·42900, is therefore adopted, and is certainly correct to 1/10,000 and probably to 1/20,000.

Nitric Oxide.—Leduc (*Séances Soc. Franç. Physique*, 1893, p. 214) determined the density of the gas in 1893, and reported it to be 1·0388 referred to air, from which the weight of a normal litre can be calculated to be 1·3429. Leduc suppressed this value in his memoirs published in 1898; therefore it can hardly be taken into account. It is evidently too high. During the investigation carried on with M. Davila we obtained very nearly the same value when using a gas which had not been purified by liquefaction and fractional distillation, so that it would appear that such a high value is due to the presence of traces of nitrous oxide in the gas.

Gray was the first to make an accurate determination of the density of this gas, using the balloon method. The gas was prepared by the action of acetic acid on sodium nitrite and potassium ferrocyanide, washed with caustic potash, dried over phosphorus pentoxide, then liquefied and fractionated at low temperatures. Six determinations, showing an extreme difference of 3/10,000, gave as the weight of the gas contained in the balloon, 0·35851. Six determinations of oxygen (extreme variation 1·3/10,000) gave 0·38228. Adopting Rayleigh's value for oxygen (1·42905) the value for nitric oxide is calculated.

$$1 \text{ litre of nitric oxide} = \frac{1{\cdot}42905 \times 0{\cdot}35851}{0{\cdot}38228} = 1{\cdot}3402 \text{ grms.}$$

On the other hand, he calculates (by private communication), from the capacity of the balloon, 1 litre of nitric oxide = 1·34011 and 1 litre of oxygen = 1·42896. Gray adopts the value 1·3402 grms. This practically conforms with that which would result if the value O_2 = 1·42900 were used.

$$1 \text{ litre NO} = \frac{1{\cdot}4290 \times 0{\cdot}35851}{0{\cdot}38228} = 1{\cdot}34015.$$

Guye and Davila have carried out three series of determinations on gas prepared from three different sources—(*a*) by the action of ferrous sulphate on nitric acid, (*b*) by decomposition by means of mercury of a solution of sodium nitrite acidified with sulphuric acid, (*c*) by the action of sulphuric acid on a concentrated solution of sodium nitrite. The method of purification was in each case the same as that employed by Gray, with the exception that the washing with caustic potash was omitted as it changes a part of the gas to nitrous oxide.

Weighings were made in two balloons of about 0·8 and 0·4 litre capacity, respectively.

Method.	No. of experiments.	Weight of 1 litre.	Extreme error. Large balloons.	Small balloons.
Mercury	6	1·3403	3/10,000	6·7/10,000
Ferrous sulphate .	6	1·3402	3/10,000	4·5/10,000
Sodium nitrite ..	2	1·3401	3/10,000	

The mean of the 14 experiments, together with the mean for each method, gives 1 litre NO = 1·3402, a value identical with Gray's, and which we hereby adopt.

Carbon Dioxide.—Rayleigh reports the density of this gas to be 1·52909 as referred to air. He gives no details as to the method, the number of determinations, or their probable error. The gas was prepared—(*a*) by the action of hydrochloric acid on marble, (*b*) by the same acid on sodium carbonate.

Leduc obtained the mean value of 1·52874 for three very concordant determinations on a gas prepared by the action of hydrochloric acid on marble. He adopts the value 1·5288, in view of the probable error due to traces of air. He also observes that the work of Regnault points to the value 1·5290.

The value for a normal litre can then be deduced as follows:—

1 litre of CO_2 = 1·29284 × 1·52909 = 1·9769 (Rayleigh).
1 litre of CO_2 = 1·29273 × 1·5288 = 1·9763 (Leduc).

Guye and Pintza have determined the weight of a normal litre directly, with a gas generated by heating sodium bicarbonate, and have found 1·9768, the mean of three experiments having an extreme difference of 0·8/10,000. The value finally accepted,—

1 litre of CO_2 = 1·9768,

agrees to about 1/20,000 with Rayleigh's value; it gives a certain weight to the lower result of Leduc, and is justified by a consideration of the probable error. In the course of the work at Geneva, the difficulty experienced in obtaining carbon dioxide entirely free from air has been constantly kept in mind, and the gas which was used in these determinations was entirely soluble in caustic potash.

Nitrous Oxide.—Leduc, Rayleigh, and Guye and Pintza have determined the density of nitrous oxide.

Leduc used the commercial liquefied gas, rectified by distillation (obtained by heating ammonium nitrate). He obtained the densities, referred to air, of 1·5304, 1·5298, 1·5301. Average, 1·5301; extreme error, 3·9/10,000. Rayleigh made his first series of determinations in 1897 with a gas obtained by the decomposition of ammonium nitrate, and purified by dissolving in water—heating to expel the gas, then drying. Five determinations with a probable error of 1·7/10,000, gave the mean value 1·52951 referred to air. In 1904 Rayleigh (*Proc. Roy. Soc.*, lxxiv., A, 181) repeated his work, using the commercial liquefied gas, purified in the same way as above, and obtained practically the same result. He then purified the commercial gas by fractional distillation at the temperature of liquid air, till the density remained constant, and obtained, as the mean of three very concordant observations, with an extreme difference of 0·8/10,000, the value 1·5297.

From this last value of Rayleigh's and from Leduc's, the following values for the weight of the normal litre may be calculated:—

1 litre of N_2O = 1·9780 (Leduc).
1 litre of N_2O = 1·9777 (Rayleigh).

Guye and Pintza have determined the weight of the normal litre directly, using the volumeter method. The gas was prepared by the double decomposition of hydroxylamine sulphate and sodium nitrite. The average of three experiments, having an extreme variation of 2·8/10,000, is—

1 litre of N_2O = 1·9774 (Guye and Pintza).

The impurities which might be found in the commercial nitrous oxide are nitrogen peroxide, air, or nitrogen. The fractionation at low temperatures might not bring about a complete elimination of these impurities, or it might be that the decomposition of hydroxylamine sulphate did not furnish a gas entirely free from air, even though the evacuation of the apparatus was repeated several times. (The washing with alkalis, as practiced commercially, is not sufficient to remove the last traces of NO_2). Under these conditions, and considering the good agreement of the separate determinations made by Rayleigh in 1904, we would adopt for the weight of the normal litre,—

1 litre N_2O = 1·9777 grms.,

which is the mean of the values obtained by Rayleigh, Leduc, Guye and Pintza.

Hydrochloric Acid Gas.—Leduc has determined the density of hydrochloric acid gas, produced by the action of sulphuric acid on sodium chloride, and dried over phosphorus pentoxide, to be 1·2692 as compared with air. There is no indication of the number of determinations or their accuracy, although he considers the last decimal doubtful. A normal litre would be 1·29273 × 1·2692 = 1·6407 grms.

Guye and Gazarian have repeated this determination with a gas prepared in the same way, but further purified by liquefaction at the temperature of liquid air and fractionation at a low temperature (see Note). A preliminary series of four determinations gave a mean of 1·6398, with an extreme variation of 9/10,000.

Note.—These fractionations were for the purpose of eliminating the volatile phosphorus compound, which, according to Richards and Wells (*Journ. Am. Chem. Soc.*, xxvii., 459) is formed when hydrochloric acid passes over phosphorus pentoxide, and which would increase the density of the gas. Our work has also shown that the desiccation of the inside walls of the balloon is accomplished only after repeated rinsings with the hydrochloric acid gas. For these reasons Leduc's value must certainly be a little too high.

Gray, who has undertaken a revision of the density of hydrochloric acid gas, reports a series of six determinations, with a probable error of 3·7/10,000, of which the mean is 1 normal litre = 1·6397. This differs from the preceding by 1/16,000. The mean of these two concordant values would be 1·63975, or, suppressing the last decimal, 1·6398. This value is given only provisionally, until further determinations shall be announced.

Ammonia Gas.—Leduc used ammonia gas obtained from a commercial ammoniacal solution called "pure," which he dried over molten caustic potash. Without reporting the number or the accuracy of his determinations, he gives the value 0·5971 as compared with air, or the weight of 1 normal litre,—

$$1{\cdot}29273 \times 0{\cdot}5971 = 0{\cdot}7719 \text{ grm.}$$

Guye and Pintza have determined the weight of a standard litre, using commercial liquefied ammonia. Assuming that Leduc's high value was due to the presence of organic bases, these experimenters previously purified the gas by leading it over red-hot quicklime, to transform the nitrogen of the organic bases into ammonia. The gas was then collected as ammonium chloride, in this form was recrystallised, and was finally set free by warming the salt with lime, and dried over long columns of recently molten caustic potash. It was entirely soluble in sulphuric acid. Five determinations (using a volumeter of 3·5 litres capacity) with an extreme error of 3·9/10,000 gave a mean value 0·77079, or in round numbers, 1 normal litre = 0·7708. Perman and Davies (*Proc. Roy. Soc.*, 1906, lxxviii., A, 34) have repeated this determination, both by the volumeter method, using a balloon of 1·7783 litres capacity, and by the balloon method, using a balloon of 0·50476 litre capacity. With the gas from a commercial ammoniacal solution, repeating Leduc's procedure, they have arrived at a value 0·7717, almost identical with Leduc's, thus proving the necessity for removing the organic bases. They then purified the gas by three different methods—(*a*) by the method of Guye and Pintza, (*b*) by decomposing with caustic potash, ammonium oxalate which had been ten times re-crystallised, (*c*) by reduction of sodium nitrite, in a caustic soda solution, by aluminium. They obtained the following results :—

Method.	No. of expts.	Weight of 1 litre.	Extreme error.
Volumeter, prepared by *a*, *b*, *c*	7	0·77085	5·2/10,000
Balloon, prepared by *a* ..	4	0·77086	0·8/10,000

The extreme error in the first series is undoubtedly due to the fact of the ammonia gas having been prepared by several methods. The authors advise the value 0·77085.

It appears from these experiments that Leduc's value cannot be considered in deciding upon a final value, as it is unquestionably affected by a constant error due to the presence of organic bases. The mean of the other three values is 1 litre of ammonia = 0·770837, which can be taken in round numbers to be—

1 litre of ammonia = 0·7708,

differing from the above by 1/19,000. It takes into account the fact that any source of error (presence of air or of organic bases, traces of moisture or condensation on the surface of the balloon) would tend toward raising the final value rather than lowering it. The mean of the determinations by Perman and Davies, using the volumeter method, which they consider most accurate, and gas prepared by method (*a*), is 0·77080 grm.

(Of the three methods, *a*, *b*, *c*, the first would undoubtedly give the purest gas. The re-crystallisation of ammonium oxalate does not assure a complete elimination of organic bases. which is likewise true of the reduction of sodium nitrite by aluminium, as the latter may contain small amounts of carbon capable of being transformed into organic bases. In the course of my work with Pintza we proved conclusively that the ammonia gas obtained by the decomposition of magnesium nitride contained traces of organic bases. Its density was 1/1935 higher than that of the gas prepared by method *a*).

Sulphur Dioxide.—Leduc reports 2·2639, referred to air, as the mean of several determinations (he does not state the number) having an extreme variation of 1·3/10,000. The gas was prepared by the action of mercury on pure sulphuric acid.

Jaquerod and Pintza, using the volumeter method (capacity of balloon = 3·5 litres) and a gas prepared by repeated fractional distillations of the liquefied sulphur dioxide of commerce, have obtained as the weight of 1 normal litre of sulphur dioxide 2·9266 grms., which represents the mean of seven determinations having an extreme difference of 1·7/10,000.

This result agrees exactly with Leduc's,—

1 litre of sulphur dioxide = 1·29273 × 2·2639 = 2·9266.

Baume, using two balloons of 0·3 and 0·5 litre capacity, respectively, has recently repeated this determination in the Geneva laboratory and obtained the same result. The gas was purified by the same method as that employed by Jaquerod and Pintza. The extreme variation was 3·8/10,000 for the large and 12·7/10,000 for the small balloon. The mean of the two series is—

1 normal litre = 2·9266.

This, therefore, is the accepted value.

Conclusions.

The various results have been tabulated as follows :—In Column I. are the values obtained by Leduc ; in Column II. those obtained by Rayleigh ; in Column III. those of various other investigators (Morley, Ramsay, Gray, Perman and Davies) ; in Column IV. those obtained in the Geneva laboratory ; in Column V. the finally accepted value ; in Column VI., the same value referred to the density of oxygen as 1. This table has been completed by the addition of the better recent determinations of other gases (Dapier, *Journ. Chim. Phys.*, 1907, v., 203, and *Arch. Sci. Phys. Nat.*, [4], xxiv., 34).

The conclusions to be drawn from the work carried on at Geneva during the last three years, compared with that accomplished in other laboratories, are as follows :—

1. The method gives results generally agreeing to at least 1/10,000, when one takes the mean of a half dozen determinations, the extreme difference between any two of which is not more than 3/10,000 or 5/10,000.

2. This agreement may be obtained even with gases as difficultly dried and purified as ammonia, hydrochloric acid, sulphur dioxide, or nitric oxide. For these it is only necessary that the balloon be well dried with dry air, and

Weight of Normal Litre (in Grms.).

Gas.	Leduc.	Rayleigh.	Others.	Laboratory. Guye.	Accepted value.	Referred to density of O_2.
O_2	(1·4288)	1·42905	1·42900 M. 1·42896 Gr.	(1·4292) J. P.	1·42900	1·00000
H_2	0·08982	(0·08998)	0·089873 M.	—	0·08987	0·062890
N_2	1·2503	1·2507	1·2507 Gr.	—	1·2507	0·87523
CO	1·2501	1·2504	—	—	1·2504	0·87502
NO	(1·3429)	—	1·3402 Gr.	1·3402 G. D.	1·3402	0·93786
Ar	—	1·7809	1·7808 R.	—	—	1·2463
CO_2	1·9763	1·9769	—	1·9768 G. P.	1·9768	1·3833
N_2O	1·9780	1·9777	—	1·9774 G. P.	1·9777	1·3840
HCl	(1·6407)	—	1·6397 Gr.	1·6398 G. G.	1·6398	1·1475
NH_3	(0·7719)	—	0·77085 P. D.	0·7708 G. P.	0·7708	0·53940
SO_2	2·9266	—	—	2·9266 J. P. 2·9266 B.	2·9266	2·0480
Air	1·2927	1·2928	—	—	1·2928	0·90469

then rinsed several times in succession with the dry gas which is to be studied. Under these conditions there is no appreciable difference between the mean obtained for a balloon of 3·5 litres capacity and one of 0·5 litre. The surface action is then negligible in work of an accuracy of the order of 1/10,000.

3. The purification of the gas is especially important. The method based on the liquefaction of the gas, followed by distillation at low temperature, is recommended.

4. It is preferable to calculate directly the weight of a litre of the gas under standard conditions, rather than to report the density as compared with that of another gas (oxygen or air). By calibrating the balloon at 0° its volume may be determined to 1/30,000—an accuracy which could be approached, in the density determination, only by a very considerable number of determinations.

5. The most probable values for the density of the gases, determined with an accuracy of the order of 1/10,000, are collected in Columns V. and VI. of the last table.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 58).

Procedure and Notes (continued).

The Aluminium Group.

Procedure 53.—Acidify the alkaline solution (P. 52) with HNO_3 (1·42), avoiding a large excess; add NH_4OH (0·96) until the mixture after shaking smells of it, and then add 2—3 cc. more. Heat almost to boiling in order to coagulate the precipitate, filter, and wash thoroughly with hot water. (White, flocculent precipitate, presence of *aluminium* (or **beryllium*); colourless solution, absence of *chromium*). (Precipitate, P. 54, or to detect beryllium, *P. 58*g* and *h*; filtrate, P. 55).

Notes.

1. The alkaline solution is acidified with HNO_3, instead of with HCl, because the latter acid might reduce chromic acid, especially if a large quantity were added, or if the acid solution were heated. A moderate excess of NH_4OH must be added in order to keep the zinc in solution, which it does because of the production of $Zn(NH_3)_4{}^{++}(NO_3{}^{-}{}_2)$; but a large excess is to be avoided, since it dissolves $Al(OH)_3$, owing to formation of $NH_4{}^{+}AlO_2{}^{-}$. The zinc is dissolved even when carbonate, phosphate, or oxalate is present.

2. Since aluminium and silica are very likely to be present in the NaOH and Na_2O_2 used as reagents, and since they may be taken up from the dishes, a blank test for these impurities should be made whenever new reagents are employed for the first time, by following P. 52 and 53 and comparing the NH_4OH precipitate with that obtained in any regular analysis. It is also well at the same time to test for zinc by acidifying the NH_4OH solution with acetic acid and following P. 57.

Procedure 54.—Dissolve the precipitate (P. 53), or a small portion of it if it is large, in 5 cc. HNO_3 (1·20). From the appearance of the precipitate estimate the number of milligrms. of aluminium which have been dissolved, and to the solution add about one-fourth as much cobalt as cobalt nitrate, using, however, not less than 0·2 mgrm. Evaporate almost to dryness in a casserole, add a drop or two of water, and soak up the solution in a small piece of filter-paper. Make a small roll of the paper, wind a platinum wire around it to form a spiral, and incinerate the paper in a small flame, finally heating the residue strongly. (Blue residue, presence of *aluminium*).

Notes.

1. This confirmatory test for aluminium should always be tried when the NH_4OH precipitate is small, for general reasons, inasmuch as the precipitation by NH_4OH of an element whose hydroxide is soluble in NaOH is not very characteristic (lead, antimony, tin, and beryllium showing a similar behaviour), but also especially to guard against mistaking SiO_3H_2 for $Al(OH)_3$, for the former substance, if not entirely removed by proper dehydration in the process of the preparation of the solution (in P. 3, Part I.), will appear at this point. A gelatinous precipitate which does not dissolve in HNO_3 indicates silica; it may be tested for by P. 5.

2. The test described in this procedure depends upon the formation of a blue compound, whose formula is not definitely known; but it is doubtless a compound of the two oxides $CoO.xAl_2O_3$, and may be simply cobalt aluminate, $Co(AlO_2)_2$. It may be formed in various ways; but the process described in the procedure seems to be the most suitable one for making the test for aluminium reliable and delicate. It is of the utmost importance to have the aluminium present in excess; for, otherwise, the blue colour is obscured by the black oxide of cobalt. In order that a small enough amount of cobalt may be added, it is convenient to use a very dilute solution of cobalt nitrate, say one that contains one-tenth of a milligrm. of cobalt per cubic centimetre.

3. When the test is properly made, the ash retains the form in which the filter-paper was rolled, and the whole mass, or a large part of it, is coloured blue. The presence of an equivalent amount of phosphate does not spoil the test. When sodium or potassium salts are also present, the ash fuses together, and the test is very unsatisfactory. For this reason the sodium salts present should be all washed out of the NH_4OH precipitate

* From the *Journal of the American Chemical Society*, XXX., No. 4.

before dissolving it in HNO_3. No other element gives a blue colour to the ash, but certain elements, especially iron, obscure the test, so that it can be applied only after other elements have been removed in the regular process of analysis. 0·5 mgrm. Al may be easily detected, and even 0·2 mgrm. after a little practice.

4. Another very good confirmatory test for aluminium consists in dissolving the NH_4OH precipitate in HCl (1·12), adding one and one-half volumes of ether, and saturating the mixture in the cold with HCl gas, as described in *P. 58*g*. Under these conditions aluminium separates in the form of the crystalline compound $AlCl_3.6H_2O$; but no other element of the aluminium group is precipitated, except chromium, which if present in quantity greater than 10—20 mgrms. gives a violet precipitate. Moreover, silica does not interfere with this test. By this process, moreover, beryllium is quantitatively separated from aluminium, and may be tested for in the filtrate.

Procedure 55.—Acidify the NH_4OH solution (P. 53) with 30 per cent acetic acid solution, avoiding an excess of more than 2 cc.

If the solution is colourless, treat it by P. 57.

If it is at all yellow, add about 10 cc. 10 per cent $BaCl_2$ solution, allow the mixture to stand for at least five minutes, and filter. (Yellow precipitate, presence of *chromium*). (Precipitate, P. 56; filtrate, P. 57).

Notes.

1. The presence of less than 0·5 mgrm. chromium as chromate in a volume of 50 cc. makes the solution distinctly yellow, and the addition of $BaCl_2$ is therefore unnecessary when the solution is perfectly colourless. It is to be avoided, since $BaSO_4$ may be precipitated and has then to be removed by filtration. In doubtful cases the colour of the solution should be compared with that of water. The colour test is, of course, not delicate by artificial light.

2. Since some sulphate may be present, the formation of a white precipitate with $BaCl_2$ does not prove the presence of chromium. Whether the precipitate is pure white or yellow should therefore be carefully noted. The yellow colour of a small $BaCrO_4$ precipitate is most apparent when the precipitate has settled or when it has been collected on the filter. If there be sufficient sulphate present to obscure the yellow colour of a little $BaCrO_4$, the confirmatory test for chromium described in the next procedure should be tried.

Procedure 56.—Pour repeatedly through the filter a 5—10 cc. portion of a mixture of 1 volume of HNO_3 (1·20) with 9 volumes of water; to the cold solution in a test-tube add about 2 cc. of ether and 1 cc. 3 per cent H_2O_2, and shake. (Blue coloration of ether layer, presence of *chromium*).

Notes.

1. This blue compound which is formed by the action of H_2O_2 on chromic acid is one of the perchromic acids. It has the formula H_3CrO_7, and appears to be an addition product of H_2O_2 and a higher oxide of chromium. It is a very unstable substance; by its decomposition oxygen is evolved and the chromium is reduced to a chromic salt. Its decomposition is greatly accelerated by an excess of H_2O_2, by the presence of much acid, and by raising the temperature. It is therefore important not to add too much H_2O_2, and to use dilute acid, as directed in the procedure. If, in dissolving the precipitate, the filter be heated with HNO_3, the paper causes the chromate to be reduced to a chromic salt; but when the cold acid is merely poured through the filter this reduction does not take place. If a green solution should be obtained, the chromium must be reoxidised with Na_2O_2 in alkaline solution before making the test. Under proper conditions 0·2 mgrm. Cr may be detected, but the test may fail with a much larger amount if the directions are not followed.

(To be continued).

CORRESPONDENCE.

CONSTITUTION OF THE CARBON MOLECULE.

To the Editor of the Chemical News.

SIR,—I am much interested in Mr. J. C. Thomlinson's suggested geometrical formula for the carbon molecule (CHEMICAL NEWS, xcviii., 37).

As I pointed out in a note appended to a later paper, one factor which would tend to diminish the discrepancy between the experimental and calculated values is the latent heat, for in the first paper (CHEMICAL NEWS, xcvii., 37) I had tacitly assumed that the heat necessary to convert solid carbon into the gaseous state at constant temperature was solely employed in splitting up complex molecules into simpler ones, an assumption which is probably wrong.

With regard to Mr. J. C. Thomlinson's thermal equivalents for the elements, it appears that I have not yet made it quite clear that the data we have at our present state of knowledge relative to the subject reduce to a set of indeterminate equations. It is possible, therefore, by making some assumption to obtain an indefinite number of sets of constants all of which give results in equal agreement with the experimental, so that this forms, in such cases, no criterion of their correctness.

The difficulty is overcome and an unjustifiable assumption avoided by using a set of complex constants, chosen so as to obtain equations which are capable of solution, such as my own system of "fundamental constants."—I am, &c.,

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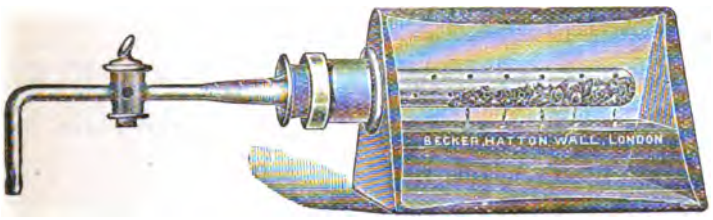

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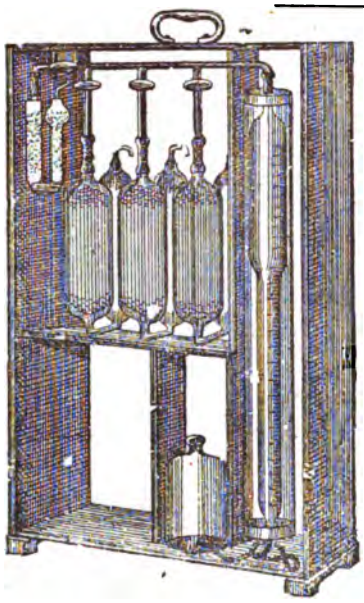

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RADIO-ACTIVE CHANGES IN THE EARTH.*

By The Hon. ROBERT JOHN STRUTT, M.A., F.R.S.

You will have already heard during the present Lecture Session from Professor Rutherford, of the recent developments of the new science of radio-activity. I venture to hope, however, that there may be room to say something of aspects of the subject not treated by him. I wish particularly to refer to manifestations of radio-activity which are observed, not in artificially prepared materials like radium, but in the rocks and minerals of the earth's crust, as we find them in nature. Let us consider, in the first place, the most conspicuous cases of this kind. The source from which radium is obtained is the mineral pitchblende. This mineral occurs in veins, like the majority of the useful metals; I may refer particularly to the mineral veins of Cornwall, so long famous as a source of tin. These veins are of the nature of cracks, running through the granite and through the slate which adjoins it. The cracks have been filled up by the various metallic ores which have been introduced by precipitation or sublimation, the exact nature of the process being somewhat obscure.

I will now show you an experiment, due to Sir W. Crookes, which illustrates the radio-activity of pitchblende in a very beautiful manner. A flat polished slab of pitchblende intergrown with a variety of other material which is not radio-active was laid face to face with a photographic plate, which was developed after the lapse of about a week of contact. The radium and other radio-active substances contained in the pitchblende have acted photographically upon the plate, while, of course, those portions of the material which are not radio-active have exerted no such action. Thus pitchblende has, as it were, taken its own portrait, which I now show you on the screen.

Pitchblende, the principal radium ore, contains, as you know, only an infinitesimal percentage of radium, the bulk of the substance being made up of oxide of uranium. Uranium is commonly spoken of as a rare metal; but terms of this kind are comparative only, and in contrast with radium, which is more than a million times scarcer, it seems common enough. Now I wish to speak for a little about this association of uranium and radium in pitchblende. Is it accidental, or has it some special significance? I hope to be able to convince you that it has.

In the early days of radium, it was common to hear the difficulty emphasised, that while there was no reason for doubting that the radium which was found in the earth had been there as long as other metals, a substance that was continually giving out energy in this way was obviously defying the greatest physical generalisation of the Nineteenth Century—the law of the conservation of energy. We cannot, however, afford to sacrifice this law so easily, and a ready mode of escape offers itself, if we suppose that a continual waste of radium is occurring. In that case it becomes necessary to suppose also that the supply is in some way replenished, for otherwise all the radium would have wasted long ago. From what material are the fresh supplies of radium derived? They must be derived from some other substance contained in the mineral where the radium is found, and there is now reason to feel sure that uranium is the substance in question.

We have convincing proof of this, in the fact that the amount of radium found in the mineral is always in direct proportion to the quantity of uranium which it contains. I should perhaps, say, to avoid misconception, that there is good reason for believing that several transitional stages exist through which uranium passes on its road to become radium. It is not necessary, however, to take into account the existence of these intermediate products in order to form a clear idea of the process by which the supply of radium is kept up. Uranium changes spontaneously though very slowly into radium, and the amount of radium produced per annum, for example, will be proportionate to the amount of uranium present. On the other hand, a certain fraction of the total amount of radium present decays per annum, and the balance of this account of profit and loss will represent the amount of radium found in the mineral at any time that we examine it. There will be no difficulty in seeing that on this theory the amount of the radium in the mineral should be proportionate to the amount of uranium, and experiment fully confirms the theory by showing that such is in fact the case. We have here a clear and distinct case of the transmutation of metals, so long unsuccessfully searched for.

Let us now come back to the pitchblende.

What was the source of metalliferous ores found in mineral veins is a very much vexed question, and no solution of it which has yet been proposed can be said to be altogether free from difficulty. One of the most plausible theories, however, supposes that the metals have been derived from the rocks by which the veins are traversed. We are not here concerned with metalliferous ores in general, but only with those which carry radio-active material. In deciding whether the granite of Cornwall can be supposed to furnish the uranium of pitchblende, it is, of course, fundamental to know whether any uranium is present in the rock. It should be said, by way of preface, that the quantity must, at best, be very small, and certainly too small for detection by the methods of chemical analysis as ordinarily applied. We have seen that uranium in nature is invariably accompanied by a proportionate quantity of radium, and as it is in practice much easier to detect minute quantities of radium than to detect the corresponding quantities of uranium, it is best to look for the former only, and to be content to infer the presence of the latter.

I have made a large number of experiments to find out how much radium there may be, not only in Cornish granite, but in a large variety of other rocks. In every case the presence of radium has been established, though only to the extent of about one-millionth part of what is found in pitchblende, and even that, it will be remembered, is not much. If we take into account the very large bulk of the granite and the very small bulk of the pitchblende veins running through it, there is no difficulty in admitting that the granite was capable of supplying the radio-active material of the pitchblende.

Granite, of course, consists of a variety of different minerals, which give it its mottled appearance. These minerals, there is no reason to doubt, have been formed in the successive stages of crystallisation of an originally molten mass. There is a mineral called zircon, of which the jacinths sometimes set by jewellers are a variety, which is present in very minute crystals in granite. These minute crystals of zircon have a very characteristic geometrical shape; a square prism terminated at each end by a pyramid, as you see in the photograph. The fact that they have this perfect shape is a proof that they have been perfectly free to assume their natural form, and have not been hampered for want of space by other minerals surrounding them. The inference is plain that zircon has been one of the first minerals to crystallise in the consolidation of granite.

I have found that this zircon is very much richer in radium than the granite generally, though, on the other hand, it is poor compared with pitchblende. It seems clear that the minerals which crystallise first take an unfair share of the radio-active elements, leaving the rest of the magma impoverished.

In the light of this observation, Professor Joly, of Dublin, has been enabled to explain a curious appearance which is seen when a section of the granite thin enough to

* A Discourse delivered at the Royal Institution, March 27, 1908.

be transparent is examined under the microscope. You see upon the screen one of Professor Joly's photographs of this appearance. This is a minute crystal of zircon, which is imbedded in a large crystal of mica. You will observe that the material surrounding the zircon for a definite distance outwards has become darkened in colour. The altered region round the speck of zircon is practically circular, and is reminiscent of a spot of grease on cloth.

Professor Joly has pointed out that this alteration in the surrounding materials must be due to the radio-activity of the zircon. That radio-active materials are capable of producing such colorations has been known from the early days of radium. You see, for instance, projected on the screen, the image of a glass bottle, in which a radium preparation has been kept. Though originally of colourless glass, it has been stained a deep purple by long continued action of radium.

It may, perhaps, be thought that this idea, though plausible, is no more than a guess. It is, however, much more than that. We know, from the investigations of Professor Bragg and Mr. Kleeman, that the α-particles of radium, which constitute the most important feature of radio-active emission, are only able to penetrate a limited and definite distance into solid materials. They then lose their characteristic properties, if, indeed, they are not altogether stopped. This distance has been measured experimentally, and Professor Joly has shown that the distance is just the same as the distance to which the alteration round the zircon crystals extends. Thus we have full quantitative confirmation of the theory which attributes it to radio-activity.

I will now pass from the discussion of a very minute phenomenon to the discussion of a large scale one. It will be familiar to many of you that, in the opinion of some, at least there is reason for changing the views which have been held for two generations concerning the earth's internal heat. We know that there is, at any rate, some radium in the earth, and that radium gives out heat. Thus it cannot be disputed that some part of the earth's internal heat must be due to this cause; the only question which remains is whether this part is large or small; whether, in fact, the earth's internal heat is chiefly to be accounted for as a small remnant of the much greater internal heat which it once possessed, or whether there is enough radio-active material in the earth to supply most of the annual loss by conduction through the crust and radiation into space.

As I mentioned before, I have made a large number of determinations of the quantity of radium in the rocks of which the superficial portions of the earth are constituted. These are found to be so rich in radium that the difficulty is not so much to account for the internal heat of the earth, as determined by underground observations of temperature, but rather to understand why it is not much hotter. I have suggested, as an explanation, that this general distribution of radio-active material, which pervades the outer parts of the earth, is in reality superficial, extending only to some moderate number of miles in depth, though no doubt much deeper than the deepest mines. I am not wholly satisfied, however, of the sufficiency of this explanation. Radium, and the series of products of which it is one, are not the only radio-active materials in the earth; there is another series, of which thorium is a member, and there is good reason to suppose that thorium is present in rocks in such quantity as to add appreciably to the evolution of heat. Taking this into account, we should probably find, if we had exact data for calculation, that the thickness of rock containing radio-active material was so small that the material of the interior would somewhere have exuded, in the course of the violent dislocations and earth movements which geology reveals to us. No material, however, appears anywhere at the earth's surface which can plausibly be regarded as representative of the unknown interior, if the suggested hypothesis is accepted. It cannot be denied that the subject is at present obscure. Possibly an explanation may be found by supposing that the activity of uranium may be arrested at high temperatures. We have at present no adequate experimental evidence on the subject. It is known that there is very little effect of this kind on radium. If, however, the activity of uranium were arrested at a high temperature, the supply of radium and all the other members of the series would fall off, and thus the aggregate heat production of the whole series might be greatly diminished.

I shall now pass to another branch of the subject. The investigations of Sir William Ramsay and Mr. Soddy have proved that there is continuous evolution of helium from the radium emanation. We have good reasons, into which, however, I do not propose to enter, for considering that the same is true of radio-active changes in general, at all events those in which there is an emission of radiation. Helium is probably evolved at each stage of the transformation of uranium, and at each stage of the transformation of thorium; and it results that the natural minerals and ores in which these elements are found, contain a store of helium, which has accumulated in them, and remains locked up in their pores.

As already mentioned, I have succeeded in determining the presence of radium in granite. Thus it becomes natural to inquire whether the corresponding amount of helium is to be found there too. Nothing of the kind had ever come under observation before, and it was therefore with some interest that I made the experiment. You see before you a vacuum tube of helium prepared from ordinary granite. The characteristic yellow glow will satisfy anyone acquainted with the appearance of a helium discharge of the presence of the gas.

The facility with which helium was detected in granite suggested further experimental problems. The undoubtedly radio-active elements are at present confined to uranium and thorium, and their respective families of descendants. Evidence has been produced, by myself among others, which suggests that lead and some other elements possess a feeble radio-activity of their own. But this evidence is somewhat equivocal. It seemed highly desirable to attack the question in a new way, and the idea suggested itself of looking for helium in the naturally occurring ores of all the elements, common and rare. This had indeed been done, to some extent, from quite a different point of view, by Sir William Ramsay and his coadjutors, in their first investigations on helium; but their observations were directed to finding a practical source of the gas, and were not carried out with anything approaching the minuteness required for the present purpose.

The upshot has been to prove the presence of helium in almost every mineral examined, and even in such unpromising materials as rock crystal, or common quartz sand. The quantity found in the various cases has varied very widely. In fact, minerals may be found having any helium content, from thorianite, which contains 10 cc. per grm., down to rock crystal, which contains about a ten-millionth part of that quantity.

I have here a small tube of helium obtained from clear, colourless rock crystal, and you will have no difficulty in seeing the characteristic yellow glow as before.

Are we to regard the helium in common minerals as due to a feeble radio-activity of the common elements? No doubt such an hypothesis is tempting, but it must be rejected. Radium is present everywhere in traces, and these traces are in general sufficient to account for the minute quantities of helium. This is illustrated in the table below, which gives in round numbers the actual amount of helium extracted from various minerals by heat, and the amount of helium reckoned relatively to the radium.

There is reason to think, as already mentioned, that the presence of thorium would constitute another source of helium. But it is believed that this complication does not produce any appreciable effect in these cases. You will see that minerals like quartz, though they contain actually only an infinitesimal quantity of either substance, still show about the same proportion of helium to radium as the

Mineral.	Helium present, c.mm. per kil.	Helium ratio, *i.e.*, ratio of helium to radium (arbitrary scale).
Normal—		
Samarskite	1,500,000	14
Hæmatite	700	9
Galena	2	17
Quartz	2	10
Abnormal—		
Beryl	33,000	954

minerals which are rich in both. We may conclude that helium is connected with radium in the poor minerals as in the rich ones.

I have, however, encountered an interesting exception to this rule in the mineral beryl. Beryl is, in all essentials, the same as emerald; the latter name is kept for stones which are of a clear deep green colour, but scientifically the distinction is of no importance. Some beryls contain enormously more helium than can be accounted for by the small traces of radium in them. Nor do they contain any appreciable quantity of other radio-active material. What view, then, can we take of the presence of helium in this mineral? It is, to me at least, difficult to believe that the gas can have been introduced from without. If not, can it have been generated from radium formerly existing in the beryl, but now exhausted? This, too, seems unlikely, for it would imply that beryls are older than other minerals, and there is no plausibility in such a theory from the geological standpoint. My own opinion is that, in all probability, an element hitherto unknown exists in the mineral, from which the helium is generated. It may be objected that, in that case, the mineral ought to be radio-active. If, however, the radiation were emitted with less than the critical velocity, we should not be able to detect it, and nothing is known to make such an hypothesis improbable.

In conclusion, I shall be well content if I have convinced you that there is still something to be learnt from careful examination of the most commonplace materials. If there is nothing new under the sun, there are, at least, unsuspected things going on inside the earth, where the sun cannot penetrate.

CHEMICAL PUBLICATIONS IN AMERICA IN RELATION TO CHEMICAL INDUSTRY.*

By W. A. NOYES.

THE American Chemical Society holds a unique place among the chemical societies of the world. It combines functions and activities which could heretofore be found only in separate organisations. It is the only large chemical society in the world which includes both those who are engaged in the application of chemistry to the industries and to chemical engineering, and those who are engaged in teaching and in researches with no immediate practical bearing, and which provides adequately for the interests of all classes of chemists. There are two reasons which justify this policy. In these days of intense specialisation one of the greatest dangers to a professional man is that he will become so intensely absorbed in the field in which he is working that he loses all interest in other lines of work, and falls out of touch with the rapid advances which his science is making in every direction. This tendency is almost as marked and fully as dangerous for those who are engaged in pure research as for those working in applied chemistry. Both classes of our members find that contact with chemists having a radically different point of view is exceedingly stimulating and useful. The advantages of an abstract journal which covers both fields are so self-evident as scarcely to require remark.

* An Address delivered at the New Haven Meeting of the American Chemical Society, July 2, 1908.

The second reason, which justifies the union of all classes of chemists in a single society, is that only in this way is it possible to provide at moderate expense those publications which are needed by every chemist. The chemists of the world seem to have realised only very dimly the advantages in economy which result from combination for purposes of publication. The fundamental principle is that for a small circulation the original cost of gathering the material and setting the type forms a large proportion of the total cost of publication. After the matter has once been accumulated and printed, the cost of printing and distributing additional copies is relatively small.

The German Chemical Society, when it began to publish officially the *Chemisches Zentralblatt*, adopted the plan that the journal was to be sent only to those who will pay for it. The result is that those who take the *Zentralblatt* must pay 9 dols. a year for it, in addition to the dues of the Society, and the journal goes to about one-half of the members. The total circulation of the *Zentralblatt* is only about 2000. The American Chemical Society in establishing *Chemical Abstracts* has adopted a different plan. While it was recognised that a few of our members might not care for the journal, it was seen that by increasing the dues of all the members, it could be sent to all at a comparatively moderate expense. Accordingly the dues were increased by 3 dols. for all members. While *Chemical Abstracts* costs, at present, between 5 and 6 dols. per member, it is securing such support among the chemists of America and of the world that the permanent success of the enterprise seems to be assured. In adopting the budget for 1907, the Council considered that if enough new members were gained during the first year to make up for those who fell out on account of the increase of dues we should do well. Instead of that the Society had a net gain of 310 members. When *Chemical Abstracts* was started in January, 1907, we had a membership of 3079. We now have 3800 members.

In establishing *Chemical Abstracts* it was recognised that the funds available would not permit of the publication of as large a journal as the *Chemisches Zentralblatt*. It has, however, approached much more nearly to the size of that journal than was anticipated, and while the abstracts in some divisions are relatively brief, it may be fairly said that *Chemical Abstracts* covers the whole field of chemistry better than any other similar publication. To illustrate, *Chemical Abstracts* gives abstracts of American, English, German, and French patents, while the *Zentralblatt* gives abstracts of German patents only. The number of abstracts in biological, mineralogical, and geological chemistry greatly exceeds the number in the same lines in the *Zentralblatt*, and the same is true of all lines of technical chemistry. The total number of abstracts, even in the first year of publication, is also greater in *Chemical Abstracts*, although the total number of pages is less.

The fields covered by *Chemical Abstracts* are so varied that at the low subscription price of 6 dols. a year it should secure many subscribers among persons whose primary interest is in other sciences than chemistry. Physicians and biologists will find much that is of value to them under biological and pharmaceutical chemistry; physicists will be interested in physical chemistry, radio-activity, and electro-chemistry; and geologists in mineralogical and geological chemistry.

As an almost necessary result of what has been stated about publication, the American Chemical Society has accepted the principle that it can afford to extend the benefits of its membership to anyone who is willing to pay the dues. The statement has recently been made that this has resulted in a membership which is largely non-professional. Such a statement is wholly misleading. While the fact that a man is a member of the Society is no guarantee that he is a trained chemist, there are very few members who are not engaged in chemical work, and I think I am safe in saying that 90 per cent of the members have had a good chemical training.

The American Chemical Society is about to establish a

Journal of Industrial and Engineering Chemistry. This journal, too, is to be sent to all members of the Society. The objection has been raised that it should be sent only to those members who are especially interested in it. Such a course does not seem wise for two reasons. First, in accordance with the ideal of the Society, which is to care adequately for the needs of all classes of chemists, we wish to continue to furnish all of our members with original papers as well as with abstracts in all fields of chemistry. Second, if we were to adopt the other plan, we could afford to give as a rebate to anyone who does not care for the journal only the amount which would be saved by printing a smaller number of copies. This amount is so small as to be scarcely an object. We seem to be justified, therefore, in adopting for the Industrial Journal the same plan which has met with so much success in the case of *Chemical Abstracts*.

The broad policies which have been adopted by our Society can succeed only on the basis of a very large membership. We need the loyal support of every American chemist. We are also securing support from nearly every civilised country in the world—from England, Germany, South Africa, Australia, New Zealand, China, Japan, Chili, Brazil, Argentina, and many others.

THE DECOMPOSITION OF CERTAIN MINERALS AND INDUSTRIAL PRODUCTS BY MEANS OF SODIUM PEROXIDE AND METALLIC SULPHIDES.*

By JAMES H. WALTON, Jun., and HERMAN A. SCHOLZ.

(Concluded from p. 64).

Sodium Peroxide Method of Decomposition applied to Manganese Determinations.

SINCE the sodium peroxide and zinc sulphide mixtures were so effective as fluxes in the lead determinations, they were tried on manganese compound.

a. Analysis of Manganese Glaze.—A manganese glaze high in silica, but containing no calcium, was first used. It was not attacked to any appreciable extent by nitric and hydrochloric acids, even after boiling for an hour. One grm. of the glaze (200-mesh sieve) was fused with sodium carbonate in a platinum crucible. The melt was dissolved, made acid with sulphuric acid (sp. gr. 1·42), and the manganese determined by Volhard's method (*Ann. Chem. Liebig*, cxcviii., 318).

	1.	2.
Per cent of MnO in the sample ..	4·57	4·55

Experiment X.

1·0 grm. manganese glaze (200-mesh sieve); 1·4 grms. zinc sulphide; 1·0 grm. sodium peroxide; 0·4 grm. potassium persulphate.

Ignited in a nickel crucible by means of magnesium ribbon. Very good fusion and apparently complete decomposition, for no residue was left on acidifying with sulphuric acid and adding a little sodium sulphite. The melt was dissolved in water, acidified with sulphuric acid (sp. gr. 1·42), and sodium bisulphite was added to reduce manganese dioxide. The sulphur dioxide was boiled off and the manganese determined by Volhard's method. Results: 4·57 and 4·55 per cent MnO.

b. Analysis of Franklinite.—The sample of franklinite contained considerable silica. As is well known, it is a very refractory mineral which cannot easily be decomposed by sodium carbonate fusion, is decomposed with great difficulty by potassium bisulphate fusion, and is decomposed by concentrated nitric and hydrochloric acids only after long boiling. A 1 grm. sample (200-mesh sieve) was boiled for two hours with nitric acid (sp. gr. 1·42) containing a small quantity of hydrochloric acid (sp. gr. 1·12). Ten cc. sulphuric acid were added and the solution boiled down to sulphuric acid fumes. It was diluted and the manganese determined by Volhard's method. Results were: 7·89 and 7·96 per cent MnO.

Experiment XI.

1·0 grm. franklinite (200-mesh sieve); 8·0 grms. sodium peroxide; 1·2 grms. zinc sulphide; 0·4 grm. potassium persulphate.

Ignited by magnesium in a nickel crucible. The mixture became liquid and red-hot, but the decomposition was not complete. The proportions of the components were varied, but it was impossible to get a complete decomposition with these ingredients. It was therefore decided to substitute for the zinc sulphide some substance which would give more heat. Iron pyrites and a small amount of magnesium powder were found to answer this requirement very well.

Experiment XII.

1·0 grm. franklinite (200-mesh sieve); 8·0 grms. sodium peroxide; 0·3 grm. powdered magnesium; 0·3 grm. potassium persulphate; 2·0 grms. pyrites.

Ignited by magnesium wire in a nickel crucible. The fused mass was extremely hot and remained liquid and red-hot for about two minutes. It gave a complete decomposition of the franklinite, but the melt dissolved very slowly, in about two hours. This was remedied by sifting 3 to 4 grms. of sodium peroxide into the liquid mass and gently rotating the crucible to mix the peroxide with the molten mass. The contents of the crucible, after cooling, dissolved easily in water in three to four minutes. The solution was made acid with sulphuric acid (sp. gr. 1·42), the manganese dioxide reduced by sodium bisulphite, and the manganese determined by Volhard's method as in the other determination.

	1.	2.
Percentage of MnO ..	7·90	7·88

c. Analysis of Ferro-manganese.—A 0·5 grm. sample of ferro-manganese which had been passed through a 100-mesh sieve was dissolved by boiling for about an hour in nitric acid (sp. gr. 1·42). Sulphuric acid was added and the solution was evaporated to sulphuric acid fumes. The manganese was determined by Volhard's method as in the franklinite determinations. Results were 56·25 and 56·23 per cent manganese.

Experiment XIII.

0·5 grm. ferro-manganese (100-mesh sieve); 8·0 grms. sodium peroxide; 0·8 grm. zinc sulphate; 0·3 grm. potassium persulphate.

Ignited by means of magnesium ribbon. A red-hot liquid mass was obtained, and the substance apparently decomposed completely. The melt dissolved with difficulty. Three to four grms. of sodium peroxide sprinkled into the fused mass produced a melt that was dissolved easily in a few minutes. The solution was acidified with sulphuric acid, the manganese dioxide reduced with sodium bisulphite, and the manganese determined by Volhard's method as before. Results: 56·18 and 56·32 per cent manganese.

TABLE II.—*Comparison of Standard and Sodium Peroxide Methods of Decomposition for Manganese Determinations.*

Sample.	Standard method, per cent MnO.	Peroxide method, per cent MnO.
Manganese glaze ..	4·57	4·58
" "	4·55	4·55
Franklinite	7·89	7·90
"	7·96	7·88
	Per cent Mn.	Per cent Mn.
Ferro-manganese ..	56·25	56·18
"	56·23	56·32

The manganese in each case was determined by Volhard's method.

* *American Chemical Journal*, xxxix., No. 7.

The Decomposition of Chrome-Iron Ore.

For these experiments a chrome-iron ore high in chromium was used. It was first analysed by fusing in a porcelain crucible with sodium peroxide, dissolving the fused mass in water, and destroying the excess of the peroxide by boiling. To the dichromate solution an excess of standard ferrous sulphate solution was added, and the excess above that necessary to reduce the sodium dichromate was determined by titrating with standard potassium dichromate. The results obtained were 47·09 and 47·12 per cent Cr_2O_3.

Experiments with a charge similar to that used in Experiment XI. showed that not enough heat is obtained with the zinc sulphide to completely decompose the chrome-iron ore. A mixture of pyrite and magnesium was found o work satisfactorily.

Experiment XIV.

0·5 grm. chrome-iron ore (through 200-mesh sieve); 8·0 grms. sodium peroxide; 0·3 grm. potassium persulphate; 2·0 grms. iron pyrites; 0·3 grm. magnesium.

Ignited in the usual manner. The fusion was very hot, but it was necessary to sift 3—4 grms. of sodium peroxide in the liquid mass to make it dissolve easily. The chromium was determined volumetrically, 47·07 per cent being found in the sample.

The Application of the Sodium Peroxide Method of Decomposition to Silica Determinations.

In order to investigate the adaptability of the sodium peroxide method to silica determinations, a blast-furnace slag was decomposed by this method and by the standard method and the percentages of silica obtained by the two methods compared. For the standard method 1·0 grm. of the slag (200-mesh sieve) was boiled for an hour with hydrochloric acid (sp. gr. 1·12). The slag appeared to be completely decomposed. It was evaporated to dryness on the water-bath and kept there for several hours after it was dry. It was moistened with hydrochloric acid, dissolved in water, and the silica filtered off. The filtrate was dehydrated a second time. The silica was ignited to constant weight in a platinum crucible and then volatilised in the usual manner, the silica being determined by difference. The results obtained were 30·06 and 30·04 per cent SiO_2.

Experiment XV.

0·5 grm. slag (200-mesh sieve); 8·0 grms. sodium peroxide; 1·2 grms. zinc sulphide; 0·3 grm. potassium persulphate.

Ignited the above mixture by means of magnesium ribbon in a nickel crucible. A good fusion was obtained. The melt dissolved easily in water and no residue was left on acidifying with hydrochloric acid. The silica was dehydrated twice and determined as before. The results were 30·02 and 29·98, as against 30·04 and 30·06 per cent by the standard method.

Qualitative Experiments.

Qualitative experiments were carried out in order to test the adaptability of this method to the decomposition of various substances.

a. Monasite.—The sodium peroxide method of fusion was tried on a sample of monazite containing 2·75 per cent ThO_2.

Experiment XVI.

1·0 grm. monazite (200-mesh sieve); 8·0 grms. sodium peroxide; 1·2 grms. zinc sulphide; 0·3 grm. potassium persulphate.

Ignited the above mixture with magnesium ribbon. The decomposition was complete, for upon acidifying with hydrochloric acid no residue was left.

b. Clays.—A brick-clay of the following composition was used:—SiO_2, 60·93; Al_2O_3, 17·93; Fe_2O_3, 8·12; MgO, 0·91; CaO, 1·35; Na_2O and K_2O, 5·01; volatile matter, 5·73 per cent. (This analysis and the one of the fire-clay were made by the chemists of the Illinois State Geological Survey).

Experiment XVII.

1·0 grm. clay (200-mesh sieve); 8·0 grms. sodium peroxide; 1·2 grms. zinc sulphide; 0·3 grm. potassium persulphate.

It was ignited by means of a magnesium ribbon. The fusion was quiet. The fused mass dissolved completely in hydrochloric acid, leaving no residue.

Experiments were made with a fire-clay of the following composition:—SiO_2, 67·37; Al_2O_3, 21·69; Fe_2O_3, 1·59; TiO_2, 1·32; loss on ignition, 8·70 per cent.

Experiment XVIII.

1·0 grm. fire-clay (200-mesh sieve); 8·0 grms. sodium peroxide; 0·3 grm. potassium persulphate; 1·2 grms. zinc sulphide.

The above mixture was ignited by means of magnesium ribbon. The melt, when taken up in water and made acid with hydrochloric acid, went into solution completely, leaving no insoluble residue. This method may be used advantageously in connection with the determination of titanium by the method described by one of the authors (Walton, "The Colorimetric Determination of Titanium," *Journ. Am. Chem. Soc.*, 1907, xxix., 481).

c. Copper Matte.—A copper matte high in copper was used in the following experiments:—

Experiment XIX.

1·0 grm. copper matte (200-mesh sieve); 8·0 grms. sodium peroxide; 0·5 grm. zinc sulphide; 0·3 grm. potassium persulphate.

This mixture was ignited in the usual manner. The mass became very hot and liquid. It was necessary to sift 3 to 4 grms. of sodium peroxide into the molten mass to make it dissolve easily. The melt, when taken up in water and acidified with hydrochloric acid, dissolved completely, leaving no residue.

d. Bauxite.—A sample of bauxite containing a considerable amount of iron and titanium oxides was used for the following experiments:—

Experiment XX.

0·5 grm. bauxite (200-mesh sieve); 8·0 grms. sodium peroxide; 1·2 grms. zinc sulphide; 0·3 grm. potassium persulphate.

Ignited by means of magnesium ribbon. The fusion was good, but the decomposition of the ore very poor. Consequently the pyrite mixture was tried.

Experiment XXI.

0·5 grm. bauxite; 8·0 grms. sodium peroxide; 0·5 grm. zinc sulphide; 1·0 grm. pyrites.

Ignited the above mixture by means of a magnesium ribbon. The fusion was extremely hot. Three or four grms. of sodium peroxide were sifted into it to make the melt dissolve easily. The melt was dissolved in water acidified with hydrochloric acid. No residue was left, which indicated complete decomposition.

e. Titanium Iron Ore.—For a titanium iron ore, high in iron and titanium, a mixture essentially the same as that used in Experiment XX. was used, but it did not decompose 0·5 grm. of the ore (200-mesh sieve).

A fusion of a mixture similar to that used in Experiment XXI. completely decomposed 0·5 grm. of the ore.

Notes and Precautions.

To ascertain the effect of the fusions on the nickel crucibles, two new crucibles were weighed, and after sixteen fusions had been made in each they were carefully cleaned and weighed again. The crucible weighing originally 18·7683 grms. lost 0·2876 grm. in sixteen fusions. A crucible weighing originally 18·6119 grms. lost 0·3363 grm. in sixteen fusions. This is an average loss

in weight of about 0·02 grm. for each fusion. When the hot oxidising nature of the fusions is considered, this is a very slight loss. The fusions were those made with the lead ores, using sodium peroxide and zinc sulphide.

It was found that a piece of twine about an inch long, soaked in alcohol and lighted, can be used for igniting the mixtures instead of magnesium ribbon. This could be used if, for any reason, magnesium was not to be introduced into the mixture.

A finely divided sample was found to be necessary for the sodium peroxide fusions. In the foregoing experiments, with the exception of the ferro-manganese and the lead slag, it was found necessary to pass the substance to be analysed through a 200-mesh sieve.

Since sodium peroxide often contains small lumps which keep the mixture from igniting or cause incomplete combustion, it is necessary to pulverise this substance before using it.

The different ingredients must be very thoroughly mixed. A partial mixing results in partial decomposition.

All the ingredients *must be perfectly dry*. The mixture is so easily ignited that if there is any moisture present it may react with the sodium peroxide and evolve enough heat to ignite the mixture prematurely.

Conclusion.

From the foregoing experiments it is evident that sodium peroxide, zinc sulphide, and potassium persulphate, when mixed in certain proportions, give a good fusion medium for the decomposition of the substances used in this investigation. In every case the new method gave results as good as those obtained by the standard method. With possibly slight modifications, it could be extended to numerous other substances, a few of which are indicated in the qualitative experiments. This method is particularly applicable to sulphide ores and to substances high in silica, such as glazes and slags. With basic substances, such as franklinite and chrome-iron ore, it is necessary to use iron pyrites to get sufficient heat. Pyrite, however, cannot be used in a good many fusions where iron is objectionable, and to fusions of that nature this method cannot be applied.

The chief advantage of the method is the quickness with which decomposition can be accomplished. The entire operation, including mixing, igniting, cooling, and dissolving the melt, takes approximately five minutes. A decomposition by acid takes from half an hour to an hour and a half or even two hours. Moreover, in most cases the nitric or hydrochloric acid must be expelled by evaporating the solution down to sulphuric acid fumes, which necessitates considerable care and usually takes half an hour longer. In the sodium peroxide method sulphuric acid may be used directly for acidifying, which does away with the tedious evaporation.

In the new method the silica present is practically all brought into solution and kept there, so that it does not clog filters and cause other annoyances. The sulphur in sulphide ores and the carbonaceous matter in slags, &c., is easily and completely oxidised by the sodium peroxide method, and the laborious oxidation by means of nitric acid is avoided.

Another advantage arises from the fact that since these fusions are made in nickel crucibles, a saving is made in the amount of platinum ware used.

Magnetic Oxides of Chromium.—Ivan Shukoff.—The decomposition of chromic acid gives a mixture of different oxides, one at least of which can exist in two states, one strongly and the other very feebly magnetic. To determine the precise composition of the magnetic oxide measurements were taken of the specific magnetism of different magnetic oxides of variable composition, to find for which composition the magnetic properties were most highly developed. Thus the formula of the magnetic oxide was found to be $2CrO_3.Cr_2O_3$.—*C. R.*, cxlvi., No. 26.

A REVISION OF THE ATOMIC WEIGHT OF LEAD.*

PRELIMINARY PAPER.—THE ANALYSIS OF LEAD CHLORIDE.

By GREGORY PAUL BAXTER and JOHN HUNT WILSON.

(Concluded from p. 65).

Method of Analysis.

THE lead chloride contained in a weighed platinum boat was first fused in a current of pure dry hydrochloric acid gas. This gas was generated by dropping concentrated sulphuric acid into concentrated hydrochloric acid, and after being washed with a saturated solution of hydrochloric acid, was passed through five towers filled with beads moistened with freshly boiled concentrated sulphuric acid to dry the gas. It has already been shown that phosphorus pentoxide may not be used for this purpose (Baxter and Hines, *Journ. Am. Chem. Soc.*, 1906, xxviii., 779). After the salt had cooled, the hydrochloric acid was displaced by dry nitrogen, and this in turn by dry air. Nitrogen was prepared by passing air charged with ammonia over red-hot rolls of copper gauze, the excess of ammonia being removed by means of dilute sulphuric acid. The gas was passed over beads moistened with a dilute silver nitrate solution and over solid caustic potash to remove sulphur compounds and carbon dioxide respectively, and was finally dried by concentrated sulphuric acid and phosphorus pentoxide. The air was purified and dried in a similar fashion. The apparatus for generating the hydrochloric acid and for purifying the hydrochloric acid and nitrogen was constructed wholly of glass with ground-glass joints. The platinum boat containing the fused chloride was next transferred to a weighing bottle without exposure to moist air, by means of the bottling apparatus, which has frequently served for a similar purpose in many atomic weight investigations in the Chemical Laboratory of Harvard College (Richards and Parker, *Proc. Am. Acad. Arts and Science*, 1896, xxxii., 59). After standing some time in a desiccator in the balance room, the weighing bottle was weighed. In most of the analyses the lead chloride was dissolved from the boat by prolonged contact with boiling water in a Jena glass flask. In the last two analyses, in order to show that no error was introduced through solubility of the glass, the solution was prepared in a large platinum retort, and was not transferred to the precipitating flask until cold.

Very nearly the necessary amount of pure silver was then weighed out and dissolved in a re-distilled nitric acid diluted with an equal volume of water in a flask provided with a column of bulbs to prevent loss by spattering. After the silver was all dissolved, an equal volume of water was added, and the nitrous fumes were expelled by gentle heating. The solution was then further diluted until not stronger than 1 per cent, and added slowly, with constant agitation, to the solution of lead chloride contained in the precipitating flask. The precipitation and handling of the silver chloride were conducted in a room lighted with ruby light. The flask was shaken for some time and allowed to stand for a few days, with occasional agitation, until the supernatant liquid had become clear. Thirty cubic centimetre portions of the solution were then removed and tested with hundredth normal silver nitrate and sodium chloride, in a nephelometer (Richards and Wells, *Am. Chem. Journ.*, 1904, xxxi., 235; 1906, xxxv., 510), for excess of either chloride or silver, and, if necessary, standard silver nitrate or sodium chloride was added, and the process of shaking and testing repeated until the amounts of silver and chloride were equivalent. The test solutions were always returned to the flask, since they contained appreciable amounts of silver chloride, and the weight of silver chloride subsequently obtained was corrected for the quantity thus introduced. Furthermore, if an excess of silver was found, a negative correction of an equivalent quantity of silver chloride was necessary.

* *Journal of the American Chemical Society*, xxx., No. 2.

THE ATOMIC WEIGHT OF LEAD.

Series I.—$PbCl_2$: 2Ag.

No. of analysis.	Sample of $PbCl_2$.	Weight of $PbCl_2$ in vacuum.	Weight of Ag in vacuum.	Weight of Ag added or subtracted.	Corrected weight of Ag.	Atomic weight of Pb. Ag=107·930, Cl=35·473.	Atomic weight of Pb. Ag=107·880, Cl=35·457.
		Grms.	Grms.	Grms.	Grms.		
1.	A	4·67691	3·63061	−0·00074	3·62987	207·179	207·079
2.	A	3·67705	2·85375	—	2·85375	207·189	207·089
3.	A	4·14110	3·21388	+0·00020	3·21408	207·173	207·073
4.	A	4·56988	3·54672	—	3·54672	207·185	207·085
5.	B	5·12287	3·97596	−0·00028	3·97568	207·201	207·101
6.	B	3·85844	2·99456	—	2·99456	207·186	207·086
7.	B	4·67244	3·62628	—	3·62628	207·189	207·089
8.	C	3·10317	2·40837	—	2·40837	207·188	207·088
9.	C	4·29613	3·33427	−0·00020	3·33407	207·202	207·102
		Average..				207·188	207·088

Series II.—$PbCl_2$: 2AgCl.

No. of analysis.	Sample of $PbCl_2$.	Weight of $PbCl_2$ in vacuum.	Weight of AgCl in vacuum.	Loss on fusion.	Weight of asbestos.	Weight AgCl from wash-waters.	Corrected weight of AgCl.	Atomic weight of Pb. Ag=107·930, Cl=35·473.	Atomic weight of Pb. Ag=107·880, Cl=35·457.
		Grms.	Grms.	Grm.	Grm.	Grm.	Grm.		
10.	A	4·67691	4·82148	0·00100	0·00021	0·00204	4·82273	207·188	207·088
11.	A	4·14110	4·26848	0·00020	0·00008	0·00180	4·27016	207·192	207·092
12.	B	5·12287	5·28116	0·00054	0·00013	0·00197	5·28272	207·181	207·081
13.	B	3·85844	3·97759	0·00035	0·00033	0·00192	3·97949	207·136	207·036
14.	C	3·10317	3·19751	0·00045	0·00014	0·00189	3·19909	207·261	207·161
15.	C	4·29613	4·42730	0·00020	0·00004	0·00268	4·42982	207·204	207·104
		Average						207·193	207·093
		Average, rejecting the least satisfactory analyses (13 and 14) ..						207·191	207·091
		Average of Series I. and II.						207·190	207·090

After the exact end-point had been obtained, about two-tenths of a grm. of silver nitrate in excess was added in order to precipitate the dissolved silver chloride, and the flask was thoroughly shaken, and allowed to stand again until the solution was perfectly clear. The silver chloride was washed, first several times with a very dilute silver nitrate solution containing four hundredths of a grm. per litre, and then eight times with pure water. It was next transferred to a Gooch crucible and dried for several hours in an electric oven, the temperature being gradually raised to 180°, and was cooled in a desiccator and weighed. In every case the moisture retained by the precipitate was determined by fusion in a small porcelain crucible. The silver chloride, dissolved in the filtrate and washings, was determined by comparison with standard solutions in the nephelometer in the usual manner. Care was taken to treat both tubes in exactly the same manner, and final readings were taken only when the ratio had become constant. Before proceeding to the nephelometer tests, however, the filtrate and washings were passed through a very small filter in order to collect a small quantity of asbestos shreds mechanically detached from the Gooch crucible. The filter was ignited and weighed, the ash being treated with a drop of nitric and hydrochloric acids in order to convert any reduced silver into chloride. In order to find out whether lead or silver nitrates were appreciably adsorbed by the filter-paper, a solution containing lead nitrate, silver nitrate, and nitric acid of the concentration of these filtrates, was passed through several small filter-papers, which were then very carefully washed. In four cases, after incineration of the papers, there was found, −0·00001, +0·00002, +0·00003, +0·00001 grm. of residue, exclusive of ash. This correction is so small that it is neglected in the calculations. In all the analyses the platinum boat behaved admirably, the loss in weight never amounting to more than a few hundredths of a milligrm.

The balance used was a short arm Troemner, easily sensitive to a fiftieth of a milligrm. The gold-plated brass weights were carefully standardised to hundredths of a milligrm. All the weighings were made by substitution with tare vessels as nearly like those to be weighed as possible.

Vacuum corrections: The values of the density of lead chloride as given by various observers range from 5·78 to to 5·805 (Landolt-Börnstein-Meyerhoffer, "Tabellen"), the mean of the more accurate determinations being 5·80. This gives rise to a vacuum correction of +0·000062 for each apparent grm. of lead chloride, the density of the weights being assumed to be 8·3. The other vacuum corrections applied were silver chloride +0·000071, and silver - 0·000031.

All analyses which were carried to a successful completion are recorded in the tables.

The close agreement of the averages of the two series is strong evidence that no constant error, such as occlusion, affects the results. Furthermore, in all, 19·55663 grms. of silver produced 25·98401 grms. of silver chloride, whence the ratio of silver to silver chloride is 132·865, a value in close agreement with the result 132·867 obtained by Richards and Wells (*loc. cit.*). The different samples, A, B, and C, all give essentially identical results.

It appears, then, that if the atomic weight of silver is taken as 107·93 (O = 16·000), the atomic weight of lead is 207·19, nearly three-tenths of a unit higher than the value now in use. If the atomic weight of silver is 107·88, a value probably nearer the truth than 107·93, lead becomes 207·09, a number still much higher than that depending upon Stas's synthesis, as is to be expected.

We are greatly indebted to the Carnegie Institution of Washington for assistance in pursuing this investigation, also to Dr. Wolcott Gibbs and to the Cyrus M. Warren Fund for Research in Harvard University for many indispensable platinum vessels.

Ergosterin and Fungisterin.—C. Tanret. — Ergosterin in ergot of rye is accompanied by a similar crystalline substance called by the author fungisterin, which appears to be present in other fungi. The two substances may be separated by a method based upon the difference between their solubilities in ether, ergosterin being the less soluble. The two substances occur in many different families of cryptogams, and apparently Gérard's generalisation is correct, viz., that all the cholesterins existing in the lower forms of plant life belong to the ergosterin group.—*Comptes Rendus*, cxlvii., No. 1.

ON THE CALCULATION OF THERMO-CHEMICAL CONSTANTS.

V.—THE CALCULATION OF THE THERMAL CONSTANTS OF AROMATIC SUBSTANCES.

By H. STANLEY REDGROVE, B.Sc. (Lond.), F.C.S.

IF we attempt to calculate the thermal constants of aromatic substances, we are beset by the difficulty of the constitution of benzene; such formulæ as those of Kekulé, Ladenburg, &c., giving inaccurate results (CHEMICAL NEWS, xcv., 301). It would be useful therefore to adopt some means of calculation, in which the question of the constitution of benzene is not introduced, where the primary object is to find out whether various groups exhibit the same thermal behaviour in aromatic as in aliphatic compounds.

This can be readily done by referring such compounds back to benzene, using the constants ζ = the molecular heat of combustion of benzene, as determined by experiment (and corrected for constant volume), and ζ' = the corresponding molecular heat of formation of benzene; whence ζ = 797·9 cals. and ζ' = −13·7 cals. (Thomsen).

This method we have already employed in calculating the fundamental thermal constants for oxygen compounds (CHEMICAL NEWS, xcvii., 253 *et seq.*); we now apply it to the other aromatic compounds, whose heats of combustion have been determined by Thomsen, not containing groups or atoms whose thermal behaviour we have not yet investigated; these compounds are three hydrocarbons and phenyl chloride.

Using the above method the molecular heats of combustion and formation of these substances have been calculated, and are exhibited, together with the differences between them and the experimental, and the percentage errors (in the case of the molecular heats of combustion) in Table I. The two oxygen compounds already considered are also given for the sake of completeness.

The agreement in all cases is perfectly satisfactory, showing that the various groups exhibit the same thermal behaviour in aromatic as in aliphatic compounds.

To facilitate reference we give in Table II. a list of the constants employed in this paper, with their respective values, together with references to the CHEMICAL NEWS wherein the method of calculating them and their respective significations will be found.

TABLE I.—*Aromatic Compounds.*

		Molecular heats of combustion.					Molecular heats of formation.			
1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.
Substance.	Formula.	Formula.	M.H.C. (Thomsen) constant volume.	M.H.C. calc.	Diff. between cols. 4 and 5.	Error.	Formula.	M.H.F. (Thomsen) constant volume.	M.H.F. calc.	Diff. between cols. 9 and 10.
			Cals.	Cals.	Cals.	Per cent.		Cals.	Cals.	Cals.
Toluene	$C_6H_5.CH_3$	$\zeta+\alpha-\beta$	953·9	956·2	+2·3	+0·24	$\zeta'+\alpha'-\beta'$	−5·3	−7·5	−2·2
Mesitylene ..	$C_6H_3(CH_3)_3$	$\zeta+3\alpha-3\beta$	1280·0	1272·8	−7·2	−0·56	$\zeta'+3\alpha'-3\beta'$	−2·4	+4·9	+7·3
Pseudocumene.	$C_6H_3(CH_3)_3$	$\zeta+3\alpha-3\beta$	1279·2	1272·8	−6·4	−0·50	$\zeta'+3\alpha'-3\beta'$	−1·6	+4·9	+6·5
Phenyl chloride	$C_6H_5.Cl$	$\zeta-\chi$	762·9	762·4	−0·5	−0·07	$\zeta'-\chi'$	−12·4	−12·0	+0·4
Anisol	$C_6H_5.O.CH_3$	$\zeta+\alpha-\omega$	934·8	933·3	−1·5	−0·16	$\zeta'+\alpha'-\omega'$	+13·8	+15·3	+1·5
Phenol	$C_6H_5.OH$	$\zeta-\omega_1$	767·6	768·2	+0·6	+0·08	$\zeta'-\omega_1'$	16·6	16·0	−0·6

TABLE II.

	M.H.C. constant. Cals.	Reference.		M.H.F. constant. Cals.	Reference.
α =	210·8	xcv., 301	α' =	21·2	xcvi., 188
β =	52·5	do.	β' =	15·0	do.
χ =	35·5	xcvii., 183	χ' =	−1·7	xcvii., 185
ω =	75·4	xcvii., 253	ω' =	−7·8	xcvii., 253
ω_1 =	29·7	xcvii., 255	ω_1' =	−29·7	xcvii., 255

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THE ESTIMATION OF IRON AND VANADIUM IN THE PRESENCE OF ONE ANOTHER.

By GRAHAM EDGAR.

THE difficulties in the separation of vanadic acid from iron were early recognised. In 1877 Bettendorf (Poggendorff's *Ann. der Phys.*, clx., 126) noted that ammonium hydroxide precipitates from a solution containing these substances a yellow vanadate of iron not entirely decomposed by an excess of the reagent. He therefore added an excess of ammonium phosphate and boiled the solution, whereby the iron was converted into ferric phosphate and precipitated as such on the addition of ammonium hydroxide, while the vanadium remained in solution. Carnot (CHEM. NEWS, lvi., 16) used repeated precipitation by ammonium hydroxide, ammonium acetate, or ammonium sulphide, this process serving to remove iron but not aluminium or chromium. According to Classen (*Am. Chem. Journ.*, vii., 349), vanadic acid may be separated from iron in ores by fusion with sodium carbonate and sodium nitrate, and extraction of the melt with water, the vanadium going into solution as sodium vanadate. According to Arnold (*Electrochemist and Metallurgist*, March-April, 1902), in the analysis of steel, a fusion of sodium peroxide and extraction with water serves to obtain the vanadic acid in solution, free from iron. Fritcherle (CHEM. NEWS, lxxxii., 258), in the analysis of carnotite, precipitated iron together with uranium and vanadium with an excess of sodium carbonate, then added sodium hydroxide and boiled for some time, the vanadic acid alone going into solution. Blum (*Zeit. Anal. Chem.*, xxxix., 156), in separating vanadium from rather large amounts of iron, as in pig-iron, states that the separation is only complete when, after precipitation of the iron as hydroxide or basic acetate, the precipitate is re-dissolved, tartaric acid added, and the iron precipitated by means of an excess of ammonium sulphide, the solution being allowed to stand for several hours. Campagne (*Ber.*, xxxvi., 3164) separates the greater part of the iron in ferrovanadium and vanadium steel by repeated extraction of the hydrochloric acid solution with ether, afterward determining the vanadium by repeated evaporation with concentrated hydrochloric acid, removal of this acid by means of sulphuric acid, and titration with potassium permanganate. He states that if it be desired to determine the iron present, the original solution, without extracting with ether, may be boiled repeatedly with hydrochloric acid to reduce the vanadic acid, the hydrochloric acid removed by means of sulphuric, and the vanadium titrated with permanganate. If then the solution be reduced with hydrogen sulphide, the excess of gas removed by boiling, and the solution again titrated with permanganate, the difference between the two titrations will give the iron present. Glasmann (*Journ. Russ. Phys. Chem. Ges.*, xxxvi., 77) effects the separation by adding potassium iodide to the sulphuric acid solution, removing the free iodine by means of sulphur dioxide, neutralising

Ferric alum in receiver (10 per cent).	$KMnO_4$, N/10×9545.		V_2O_5.			Fe_2O_3.		
	I.	II.	Taken.	Found.	Error.	Taken.	Found.	Error.
Cc.	Cc.	Cc.	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.
35	31·90	58·02	0·1136	0·1137	+0·0001	0·1437	0·1436	- 0·0001
35	31·90	58·04	0·1136	0·1138	+0·0002	0·1437	0·1435	- 0·0002
35	31·85	58·00	0·1136	0·1138	+0·0002	0·1437	0·1433	- 0·0004
35	31·90	58·00	0·1136	0·1136	—	0·1437	0·1437	—
20	25·30	38·35	0·0568	0·0568	—	0·1437	0·1423	−0·0004
20	25·29	38·30	0·0568	0·0566	- 0·0002	0·1437	0·1433	0·0004
20	15·98	29·02	0·0568	0·0568	—	0·0719	0·0721	+0·0002
50	38·50	77·60	0·1704	0·1702	−0·0002	0·1437	0·1442	+0·0005
50	38·45	77·60	0·1704	0·1704	—	0·1437	0·1438	+0·0001
50	38·45	77·58	0·1704	0·1703	−0·0001	0·1437	0·1439	+0·0002
35	22·50	48·60	0·1136	0·1136	—	0·0719	0·0720	+0·0001
35	22·50	48·60	0·1136	0·1136	—	0·0719	0·0720	+0·0001
35	22·45	48·58	0·1136	0·1137	+0·0001	0·0719	0·0716	- 0·0003
20	15·97	29·07	0·0568	0·0570	+0·0002	0·0719	0·0718	- 0·0001

with potassium hydroxide, and precipitating the iron by means of a mixture of potassium iodide and iodate. The liberated iodine is removed by sodium thiosulphate, the ferric hydroxide filtered off, and the vanadium estimated in the filtrate.

Among other methods of separation may be mentioned that of Classen (*Ber.*, xiv., 2771), who precipitates the iron electrolytically from a solution of the double oxalates, and Myers (*Journ. Am. Chem. Soc.*, xxvi., 1124), who deposits the iron, using a mercury cathode, the vanadium in both cases remaining in solution.

In view of the difficulties attendant upon the separation of iron and vanadium, and in view of the increasing importance of such substances as ferrovanadium and vanadate of iron, composed almost entirely of these elements, it has been thought advisable to present a method by which vanadium and iron may be estimated in the presence of each other.

If a solution containing vanadic acid and iron be reduced by means of sulphur dioxide, the reoxidation by potassium permanganate proceeds according to the equation—

$$5V_2O_4 + 10FeO + 4KMnO_4 = 5V_2O_5 + 5Fe_2O_3 + 2K_2O + 4MnO \quad (1).$$

If this solution, after titration, be passed through a column of amalgamated zinc in the Jones reductor, the receiving flask being charged with a solution of ferric sulphate, the reduction is carried, in the case of vanadic acid, to the condition of V_2O_2 (Gooch and Edgar, *Am. Journ. Sci.*, xxv., 233), and the re-oxidation by permanganate proceeds according to the equation—

$$5V_2O_2 + 10FeO + 8KMnO = 5V_2O_5 + 5Fe_2O_3 + 4K_2O + 8MnO \quad (2).$$

The difference in the number of cubic centimetres of permanganate used in the first and second titrations is evidently used in oxidising the vanadium from the condition of V_2O_2 to V_2O_4, and multiplied by the factor 0·00456 (for exactly N/10 solutions), gives the amount of vanadic acid present. This being known, the iron present may be calculated from the amount of permanganate used in either titration.

In the experiments in Table I. three standard solutions were used, viz. :—

1. A solution of potassium vanadate, slightly acidified with sulphuric acid, and containing 11·36 grms. of vanadic acid to the litre. This solution was standardised by the method of Holverscheit (Inaugural Dissertation, Berlin, 1890).

2. A solution of ferric alum, slightly acid, with sulphuric acid and containing 14·37 grms. of ferric oxide to the litre. This solution was standardised by the very accurate method of Newton (*Am. Journ. Sci.*, xxv., 343).

3. An approximately tenth normal solution of potassium permanganate (3·16 grms. per litre), standardised against a N/10 solution of arsenious oxide.

The details of manipulation were as follows :—Measured portions of the ferric sulphate solution were mixed with portions of the solution of vanadic acid, and a current of sulphur dioxide was passed through the slightly acid mixture until the colour had changed from red into green, and finally into a clear blue. A few centimetres of dilute sulphuric acid were then added, and the solution heated to boiling, the current of sulphur dioxide being replaced by one of air-free carbon dioxide. When the last traces of sulphur dioxide had been removed, the flask was cooled in running water, the atmosphere of carbon dioxide being maintained, and when thoroughly cool, titrated with potassium permanganate until the colour had changed from blue into yellowish green. The solution was then heated to 70—80° and the titration completed at that temperature. The solution, having now a volume of 100—150 cc., was passed through a column of amalgamated zinc in a long Jones reductor, being preceded by 150 cc. of hot dilute (2½ per cent) sulphuric acid and followed by 100 cc. of dilute acid and finally 200 cc. of distilled water. The receiving flask, containing an excess of ferric sulphate, was kept cool by means of running water, and its contents, after the addition of syrupy phosphoric acid to remove the colour of the iron, were titrated with permanganate until the colour had changed from bluish green to yellow, and the colour of the permanganate began to be persistent and destroyed only by shaking. The flask was then heated to 70—80° and the titration completed in the hot solution.

The results given in the table show that iron and vanadium may be readily estimated in the presence of each other by two oxidations with potassium permanganate, following reduction first with sulphur dioxide and last with amalgamated zinc, under the conditions described above.

In conclusion, the author desires to thank Prof. F. A. Gooch for advice given in the course of the work.—*American Journal of Science*, xxvi., July, 1908.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 72).

Procedure 57.—Warm the acetic acid solution (P. 55 or *P. 58*f*) to 50° or 60°, saturate it in a small flask with H_2S, cork the flask, and allow it to stand for five or ten minutes if no precipitate separates at once. (White flocculent precipitate, presence of *zinc*). Filter through a double filter (two filters folded together), wash once with a little water. Reject the filtrate.

* From the *Journal of the American Chemical Society*, xxx., No. 4.

To confirm the presence of zinc, pour a 5—10 cc. portion of HNO_3 (1·20) two or three times through the filter containing the H_2S precipitate. To the solution add an amount of cobalt as cobalt nitrate equal to about one-fourth of the amount of zinc estimated to be present, using, however, not less than 0·2 mgrm. cobalt. Evaporate in a casserole almost to dryness to expel the acid, neutralise with 10 per cent Na_2CO_3 solution, and add about 0·5 cc. in excess. Evaporate to dryness, ignite gently until the purple colour due to the cobalt disappears, and allow the casserole to cool. (Green colour, presence of *zinc*).

Notes.

1. ZnS precipitates more rapidly, and in a somewhat more flocculent form, from a warm solution. Very small quantities of zinc (less than 1 mgrm.) may be missed unless a short time be allowed for the precipitate to coagulate; but, since sulphur may then separate, the appearance of a white turbidity is not sufficient proof of the presence of zinc. The precipitate may be allowed to settle, in order that the amount of zinc present may be better estimated. A double filter is used, since the ZnS is apt to pass through the filter.

2. The immediate formation of a white flocculent precipitate with H_2S in acetic acid solution is so characteristic as to be a sufficient test for zinc. Manganese is the only other element of this group that forms a light coloured sulphide; and this, owing to its greater solubility in water, does not precipitate from an acetic acid solution. The confirmatory test described in the last paragraph of the procedure is, however, useful when only a small non-coagulating precipitate, which may be sulphur, results, or when, owing to the presence of a small quantity of other elements, the precipitate is dark coloured.

3. The green compound obtained in the confirmatory test is doubtless a compound of cobalt and zinc oxides, perhaps cobalt zincate, $CoZnO_2$. The conditions under which the zinc and aluminium compounds of cobalt are formed are very different. As we have seen, the aluminium compound is formed only at very high temperatures, and the test is not at all delicate in the presence of a salt of an akali element. On the other hand, the zinc compound is obtained at comparatively low temperatures, and the presence of an alkali is essential. Excess of cobalt must, of course, be avoided, for the black cobalt oxide completely obscures the green colour. A larger proportion of cobalt than is recommended in the procedure may be added without danger, but the test is very satisfactory even when a large excess of zinc is present.

**Procedure* 58*a*.—To the filtrate from the Na_2O_2 precipitate (P. 52), add HNO_3 (1·20), keeping the solution cool, until it reacts slightly acid and any precipitate just re-dissolves upon shaking. (See Note 1). Dilute the solution to 100 cc. and transfer it to a strong 200 cc. bottle. Add solid $NaHCO_3$, a little at a time, until the mixture after shaking no longer turns blue litmus-paper red at once; finally add 1·0—1·5 grms. solid $NaHCO_3$ (weighed out roughly). Close the bottle with a tightly fitting cork, wire it in, wrap a cloth around the bottle, place it in a vessel of warm water, and boil the water gently for twenty or thirty minutes. Cool the bottle to at least 50° (best by slowly adding cold water to the bath), remove the cork, filter at once, and wash, using suction if the precipitate is large. Filtrate, *P 58*b*; precipitate, *P. 58*f*).

Notes.

1. If up to this point in the analysis there has been no indication of any of the elements that are to be tested for in the alkaline solution, time may often be saved by determining before treating with $NaHCO_3$ whether any of them are present by proceeding as follows:—To one-fourth of the solution, which has been acidified with HNO_3, add NH_4OH (0·96) in small excess, note whether a precipitate forms, and then add a few drops of $(NH_4)_2S$ solution. Even if there is no precipitate, vanadium may be present; to test for this, add 3 cc. NH_4OH (0·90) and completely saturate with H_2S (see *P. 51*a*). If the results of these tests show that any of these elements are present, treat the remainder of the HNO_3 solution by the regular procedure. If, on the other hand, the results are negative, no further treatment is necessary.

2. The alkaline solution is kept cold during the neutralisation and an excess of HNO_3 is avoided because chromate in the presence of H_2O_2 and an acid is rapidly reduced to a chromic salt, especially when the solution is hot. This reduction, if complete, would prevent the detection of chromium in the subsequent test. H_2O_2 will, to be sure, not be present, since Na_2O_2 is very rapidly destroyed by the boiling of the alkaline solution in P. 52, except when uranium is also present; in this case the peruranate which is formed by the treatment with Na_2O_2 is not decomposed upon boiling, but breaks up into a uranyl salt and H_2O_2 upon acidification.

3. The success of this separation depends upon securing the proper concentration of the $NaHCO_3$. Since the $NaHCO_3$ may be at first used up in precipitating zinc, aluminium, and beryllium as well as in neutralising the free acid, the weighed amount of $NaHCO_3$ is added only after the solution ceases to react distinctly acid. A much larger concentration of $NaHCO_3$ than 1·0 to 1·5 per cent would not prevent the complete precipitation of zinc or of aluminium, but it would interfere with that of beryllium. Thus with a volume of 100 cc. the precipitation of the beryllium is complete when the concentration of the $NaHCO_3$ is 1 per cent., and 1 mgrm. can usually be detected when it is 2 per cent, but 3 mgrms. remain dissolved in a 3 per cent solution, about 15 mgrms. in a 5 per cent solution, and about 150 mgrms. in a. 10 per cent solution. A smaller concentration of $NaHCO_3$ and a smaller volume than 100 cc. are avoided, in order to prevent as far as possible the precipitation of uranyl vanadate, which may otherwise occur when large quantities of uranium and vanadium (about 100 mgrms. of each) are simultaneously present. The presence of phosphate or oxalate (or chromate) does not cause the precipitation of uranium, nor otherwise interfere with the analysis. When only small amounts of the elements of this group are present, the separation can be made in a smaller volume, care being taken that the concentration of the $NaHCO_3$ rather than the quantity taken be that prescribed.

4. If it is desired, the separation may be made in an open flask; but in this case the solution must not be boiled and the $NaHCO_3$ solution should not be stronger than 1 per cent. The process is then best carried out by digesting the mixture on a water-bath for twenty to thirty minutes in a flask covered with a watch-glass. Under these conditions neither zinc nor beryllium dissolve in significant quantity; but 1 to 2 mgrms. of aluminium may be completely dissolved. The mixture must not be boiled, for a larger amount of aluminium may then dissolve (as much as 5 mgrms. on one minute's boiling).

5. When a large amount of aluminium or beryllium is present, 2 to 5 mgrms. of uranium may be carried down almost completely in the $NaHCO_3$ precipitate, so that in this case uranium has to be tested for in the analysis of the precipitate.

6. In a dilute solution of $Na+HCO_3-$ saturated with CO_2, the hydrogen ion and hydroxyl ion concentrations are both very small and are nearly the same as in pure water. AlO_3H_3, basic $BeCO_3$, and $ZnCO_3$ are completely precipitated because the solubility of these substances is not much increased through removal of the $OH-$ or $CO_3=$ by combination with $H+$ with formation of H_2O or HCO_3- (leaving the cations $Al+++$, $Be++$, or $Zn++$ in the solution) or through removal of the metal ions by combination with the $OH-$ with the formation of AlO_2-, $BeO_2=$, or $ZnO_2=$. The higher hydroxides

of chromium and vanadium (chromic and vanadic acids) are soluble polybasic acids, which are so much more highly ionised than H_2CO_3 that they displace it from its salt $NaHCO_3$, forming mainly sodium hydrogen chromate or vanadate. The fact that uranium is not precipitated, even though $Na_2U_2O_7$ is difficultly soluble, may be due to the formation of a complex sodium uranyl carbonate.

(To be continued).

CORRESPONDENCE.

THE THERMO-CHEMICAL RELATIONS OF CARBON, HYDROGEN, AND OXYGEN.

To the Editor of the Chemical News.

Sir,—With reference to my letter to the Chemical News (vol. xcviii., p. 46) I have reconsidered the foundations of any theory of themo-chemical equivalence.

Any difficulty in notation which must occur when symbols currently in use are altered in any way whatsoever leads me to point out that, in my earlier paper (Chemical News, xciii., 37), I state that carbon has a divalent function in acetylene. This would, as the context shows, indicate that carbon in carbon monoxide has the same heat value as one carbon atom in unsaturated hydrocarbons where there is an unsaturated carbon valency represented by two bonds.

With this reservation I append the accompanying table for unsaturated hydrocarbons.

	Heat of formation (cals.).	
	Experiment.	Calculated.
Propylene	3·220	4·090
Tri-methylene ..	−3·470	−4·080
Isobutylene	10·660	10·600
Allylene	−39·950	−42·370
Di-propargyl ..	−96·040	−97·780

—I am, &c.,

J. C. Thomlinson.

6, Villa View, Low Fell, Gateshead,
August 4th, 1908.

CONSUMPTION OF SODIUM NITRATE.

To the Editor of the Chemical News.

Sir,—Will Dr. Franks kindly explain the data in the Chemical News, vol. xcvii., p. 289, how it is possible that Europe absorbs only about 340,000 tons of sodium nitrate, while Germany alone imports over 500,000 tons yearly?—I am, &c.,

Dr. A. Batez.

Plzen, Bohemia.

NOTICES OF BOOKS.

Catalogue of Lewis's Medical and Scientific Circulating Library. New Edition, revised to the end of 1907. London: H. K. Lewis. 1908.

The catalogue of this circulating library shows that the volumes it comprises have been well chosen, and its subscribers will find that it gives them every opportunity of keeping their knowledge up-to-date. The second part of the catalogue containing a classified index of subjects with the names of the authors who have treated them is a great convenience. For the benefit of book-buyers the published price of each book is stated, and subscribers to the library are given special facilities for obtaining second-hand copies of various works.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Academie des Sciences. Vol. cxlvii., No. 1, July 6, 1908.

Triboluminescence of Racemic Compounds.—D. Gernez.—The author has investigated the question of the possibility of the existence of a relation between the molecular constitution of certain substances and their triboluminescence, paying special attention to Tschugaeff's generalisation that when two constituents are triboluminescent the racemic compound formed by their union does not possess the same property. He has studied the tartrates more particularly, and the general conclusions he has drawn do not confirm Tschugaeff's view, and point to the fact that there is no connection between the triboluminescence of substances and their symmetrical or asymmetrical constitution.

Action of Metallic Oxides (Reducible) upon Primary Alcohols.—Paul Sabatier and A. Mailhe.—Certain reducible oxides, *e.g*, Sb_2O_5, Sb_2O_3, Bi_2O_3, react with primary alcohols below 400°, giving water and aldehyde; neither gas nor acid is formed, and a semi-metallic mass is left. When the oxide is reduced more readily the aldehyde is partially oxidised, giving either the corresponding acid or water and carbon dioxide. Very often the metal or lower oxide which is formed catalytically decomposes the primary alcohol. Some of the oxides which are reduced by alcohol have a catalytic action on the latter, which lasts as long as any of the original oxide is left.

Relation between the Magnetic and Chemical Properties of Complex Salts containing Iron.—P. Pascal.—The determination of the magnetic susceptibility of different solutions of ferric chloride, $FeCl_3$, potassium ferricyanide, $Fe_2(CN)_{12}K_6$, sodium ferripyrophosphate, $Fe_2(P_2O_7)_3Na_6$, and ferrimetaphosphate, $Fe_2(PO_3)_{12}Na_6$, and ammoniacal ferric phosphate, shows that in this family of complex compounds of the same type the degradation of the chemical properties of iron, from the point of view of analysis, is accompanied by a parallel degradation of the magnetic properties. If the ferric complex is destroyed by an acid it is seen that the return towards the ferric state is accompanied by an increasing paramagnetism.

New Iodide of Titanium, TiI_2.—Ed. Defacqz and H. Copaux.—To prepare the new lower iodide of titanium two boats, one of which contains the tetraiodide and the other mercury, are heated side by side in a porcelain tube through which a current of dry hydrogen is passed. Thus a sublimate is formed which consists of crystals of the di-iodide. This substance is black and shining, infusible, not volatile, and very hygroscopic. It is not reduced by hydrogen at a dark red heat, but yields amorphous titanium at a white heat. The chloride is obtained when it is gently heated in a current of chlorine. Oxygen decomposes it into iodine and titanic acid. Sulphur and phosphorus react upon it at their melting-points, giving the corresponding compounds. Nitric and sulphuric acids attack it violently in the cold, setting free iodine. Gaseous ammonia gives an addition product.

Heat of Neutralisation of Picric Acid by different Aromatic Bases in Benzene.—Léo Vignon and M. Evieux.—From the heat of neutralisation of various picrates in benzene it may be deduced that the formation of salts may take place, but the formation differs from that which occurs in water. Since benzene possesses practically no ionising power it is specially valuable for use in the thermic study of the formation of certain salts. The presence of several NO_2 groups in the radical combined

with OH makes the acid function very strong. Picric acid does not behave like an acid towards certain bases such as *o*-nitraniline. Some other substances, for instance trichloracetic acid, in which the acid character is strengthened by the substitution of 3Cl for 3H, behave in the same way as picric acid.

Direct Transformation of Borneol into Campholic and Isocampholic Acids.—Marcel Guerbet.—When borneol is heated with freshly fused potash it is transformed nearly quantitatively into campholic and isocampholic acids as shown by the equation $C_{10}H_{18}O + KOH = C_{10}H_{17}KO_2 + H_2$. The acids in the alkaline solution may be separated by adding sulphuric acid till the liquid is feebly acid, then passing in carbon dioxide and cooling with ice-water. The campholic acid is thus precipitated. The isocampholic acid can be separated by adding sulphuric acid to the filtrate, and it may be purified by crystallising its calcium salt in absolute alcohol. This transformation of borneol into campholic acid is interesting, because it is probably the type of the action of potash at high temperatures upon secondary cyclanols.

Preparation of Benzoylacetic Ethers.—A. Wahl.—When ethyl benzoate and sodium are brought together in anhydrous ether three reactions occur simultaneously:—Sodium acts upon the acetic ether, giving ethylacetylacetate; sodium acts upon the benzoic ether, giving benzoïn; and finally sodium acts upon the mixture of the two ethers, giving ethyl benzoyl acetate. In order that this last may be the predominating reaction care must be taken that the benzoic ether is not in contact with an excess of sodium, and also that the metal is not present in an excess of acetic ether. The author has also shown that the condensation of methyl benzoate and acetate readily yields methyl benzoylacetate, from which the ethers of benzoylacetic acid with higher alcohols can easily be prepared.

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The MEDICAL SESSION will be opened on MONDAY, the 12th OCTOBER, 1908.

WINTER COURSES.

Zoology, Elementary, 9 a.m.; and Zoological Laboratory, Elementary, 11 a.m. or 3 p.m.—Professor GRAHAM KERR, M.A.
Chemistry, 10 a.m.; and Chemical Laboratory, 10 a.m. to 4 p.m.—Professor FERGUSON, M.A., LL.D., F.S.A.
Organic Chemistry, Elementary.—T. S. PATTERSON, Ph.D.
Anatomy, Junior, 11 a.m.; Senior, 1 p.m.; and Practical Anatomy, 9 a.m. to 4.45 p.m.—Professor CLELAND, M.D., LL.D., D.Sc., F.R.S., and Demonstrators.
Applied Anatomy (Jan.).—ROBERT KENNEDY, M.A., D.Sc., M.D.
Physiology, 12 noon; Practical Physiology; and Physiological Laboratory.—Professor NOEL PATON, B.Sc., M.D., and Assistants.
Physiological Chemistry.—E. P. CATHCART, M.D.
Psychological Physiology.—HENRY J. WATT, M.A., Ph.D.
Materia Medica and Therapeutics, 11 a.m.; and Pharmaceutical Laboratory.—Professor STOCKMAN, M.D.
Pathology, Systematic, 3 p.m.—Professor MUIR, M.A., M.D.
Surgery, 12 noon.—Prof. Sir WILLIAM MACEWEN, M.D., LL.D., F.R.S.
Midwifery, 2 p.m.—Professor MURDOCH CAMERON, M.D.
Practice of Medicine, 11 a.m.—Professor GEMMELL, M.D.
Clinical Medicine, 9 a.m.—Professor GEMMELL, M.D.
Clinical Surgery, 9.15 a.m.—Professor Sir HECTOR C. CAMERON, M.D., and Professor Sir WILLIAM MACEWEN, M.D., LL.D., F.R.S.
Public Health, 12 noon, four days weekly; Public Health Laboratory, 10 a.m. to 4 p.m.—Professor GLAISTER, M.D., D.P.H. (Camb.).
Ear, 4 p.m., Mon. (University); Practical Instruction as may be arranged.—THOMAS BARR, M.D.
Throat and Nose, 4 p.m., Thurs. (University); Tues. or Fri. (Western Infirmary). JAMES WALKER DOWNIE, M.B.

SUMMER COURSES.

Commencing on TUESDAY, 20th APRIL, 1909.

Physics, 11 a.m. JAMES G. GRAY, B.Sc.
Practical Physics, 9 to 11 a.m., or 1 to 3 p.m.—Professor GRAY, M.A., LL.D., F.R.S.
Botany, 8 a.m.; and Botanical Laboratory, 9 a.m to 4 p.m.—Professor BOWER, Sc.D., F.R.S.
Clinical Medicine, 9 a.m.—Professor GEMMELL, M.D.
Clinical Surgery, 9.15 a.m.—Professor Sir HECTOR C. CAMERON, M.D., and Professor Sir WILLIAM MACEWEN, M.D., LL.D., F.R.S.
Chemical Laboratory, 10 a.m. to 4 p.m.—Professor FERGUSON, M.A., LL.D., F.S.A.
Organic Chemistry, Elementary.—T. S. PATTERSON, Ph.D.
Practical Anatomy, 9 a.m. to 4 p.m.—Professor CLELAND, M.D., LL.D., D.Sc., F.R.S., and Demonstrators.
Embryology, 11 a.m.; and Embryological Laboratory, 9 a.m. to 4 p.m. JAMES F. GEMMELL, M.A., M.D.
Applied Anatomy.—ROBERT KENNEDY, M.A., D.Sc., M.D.
Practical Pharmacy, 12 noon.—Professor STOCKMAN, M.D.
Practical Physiology, 8 to 10 or 9 to 11 a.m.; and Physiological Laboratory.—Professor NOEL PATON, B.Sc., M.D., and Assistants.
Medical Jurisprudence and Public Health, 11 a.m.; and Public Health Laboratory.—Professor GLAISTER, M.D., D.P.H. (Camb.).
Practical Pathology, 1 to 3 p.m.—Professor MUIR, M.A., M.D.
Operative Surgery, 12 noon, with practical work at other hours.—Professor Sir WILLIAM MACEWEN, M.D., LL.D., F.R.S.
Diseases of Women, 2 p.m., Tu., Th., Fri.—Professor MURDOCH CAMERON, M.D.
Insanity, 12 noon, Wed. (University); 10 a.m., Sat. (Gartnavel).—LANDEL R. OSWALD, M.B.
Ear, 4 p.m., Mon. (University); Practical Instruction as may be arranged.—THOMAS BARR, M.D
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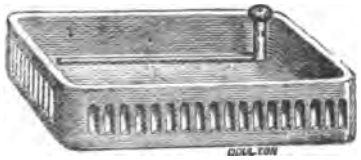

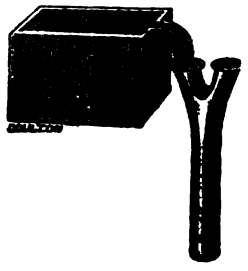

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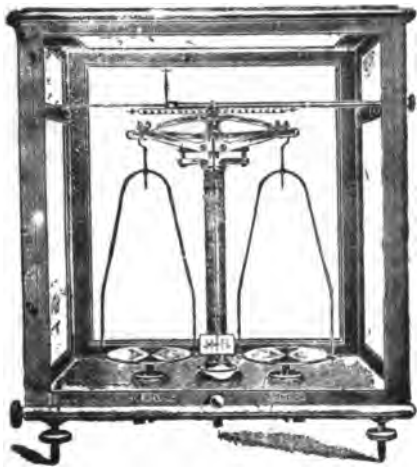

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THE CHEMICAL NEWS.

Vol. XCVIII., No. 2543.

A NEW THEORY OF MOLECULAR VOLUMES.

By GERVAISE LE BAS, B.Sc.

Introduction.

It is more than fifty years (1855) since Kopp applied his additive theory of molecular volumes to organic compounds at the boiling-point. Starting from the fundamental observation that the volume of the homologous increment, $CH_2 = 22 = constant$ at this point, he was led to the conclusion that the molecular volumes of compounds under such conditions are equal to the sums of their atomic volumes.

Subsequent observers have found that the theory of molecular volumes cannot be so simply stated, for—(*a*) the volume of CH_2, and by inference the atomic volumes, are not quite constant (Gartenmeister and Schiff; *cf.* also Young, in British Association Address, Cambridge, 1904); (*b*) it is also evident that the volumes of compounds possessing the same composition and nearly the same constitution, as, for instance, the isomeric dihalogen organic compounds (Städel), do not possess quite the same volumes at the boiling-point.

These and similar observations show that a simple additive theory does not hold good. It then becomes necessary to decide whether the above limitations require an abandonment of this view of molecular volumes, as has been done by Traube, or whether they are to be considered as due to constitutive influences.

That there is a great deal to be said in favour of an additive theory is evident from Kopp's own observations, as well as from the wealth of material that has since been accumulated, but especially by Thorpe's researches, which include determinations of molecular volumes of compounds which are usually regarded as inorganic (*Journ. Chem. Soc.*, 1880). Ostwald ("Lehrb. der Allg. Chem.," 2nd Edit., 1891, p. 356), who has made a thorough epitome of the material, has shown that simple additive relations exist, with some very considerable deviations, which are to be ascribed to the influence of constitution.

The present theory of molecular volumes is based upon a number of propositions which are shown to be supported by experimental evidence. The *first* is as follows:—

I. *The molecular volumes of chemical compounds under corresponding conditions are entirely functions of their chemical composition and constitution.*

This proposition may be inferred from Van der Waal's equation of condition. According to this, $V_k = 3b = 3V_o$; that is, the molcular critical volume is equal to three times the constant *b*, which represents the molecular volume at absolute zero. *b*, that is V_o and therefore V_k, are measures of the space occupied by the molecules, and thus similar volume relations hold under all these conditions, and also more or less under conditions that are equally reduced, as, for example, at equal fractions of the critical pressures. The probability, however, is that V_o is $\frac{1}{4}$ rather than $\frac{1}{3}$ of V_k; but this circumstance of course does not affect the volume relations.

The *second* principle is—

II. *That the molecular volumes under corresponding conditions are partly additive and partly constitutive.*

Kopp advocated an additive theory pure and simple, but observation shows that it is not true. Nevertheless, the additive principle is to a large extent valid. It will be necessary, in the first place, to correct Kopp's atomic volumes, especially those of the fundamental atoms C and H. This correction has the effect of allowing the question of influence of constitution to be more readily discussed, for then a relation is discoverable between differences in volume and the differences in constitution which are known to exist between the members of the different classes of compounds.

So far, the effect of constitution has not been very precisely defined nor studied with any degree of completeness.

Almost the only generalisation of importance coming under this head is—

That the volumes of compounds diminish more and more as the shape of the molecules tend towards the spherical.

It is, however, difficult to understand why such a relatively small difference in the constitution of such compounds as the normal and iso paraffin hydrocarbons, for example, should be measurable in terms of volume, while the very great constitutive differences which exist between the hydrocarbons hexane, benzene, naphthalene, and anthracene should not be noticeable in their molecular volumes. It is evident from the following that there are considerable differences between the volumes of compounds possessing the same composition which can only be ascribed to differences in constitution.

Thus a difference of 12·5 exists between the volumes of *heptylene* and *hexahydrotoluene*, both of which possess the empirical formula C_7H_{14}, and also a difference of 13·2 between the volumes of *cymene* and *hexahydronaphthalene* of formula $C_{10}H_{14}$. These differences are not accounted for on Kopp's hypothesis—although he was aware of their existence, and thus there appears to be in it a deficiency of capital importance.

The *constitutive influences* which might conceivably affect the molecular volumes of compounds are:—

a. Change of valency of one or more of the constituent atoms.
b. The complexity of the compounds.
c. The arrangement of the atoms in the molecule.

(No notice is here taken of association, as it does not appear to affect molecular volume very considerably, if at all).

A consideration of the influence of (*a*) on the molecular volume is reserved till later.

It appears that the complexity of a compound is a factor influencing the volume to a slight extent. This is shown by the fact that the volume of the homologous increment CH_2 varies slightly from compound to compound. Consequently, the atomic volumes may be supposed to differ. It nevertheless follows that the apparent volume of CH_2 is not usually the true one, for its magnitude depends on the changes which may occur from compound to compound, influencing every atom proportionately, and not only the increment CH_2.

Young has shown (British Association Address, Cambridge, 1904) that, under all conditions, the apparent volume of CH_2 is not constant. In Table I. it is found that the molecular volumes and the differences Δ are given under the following conditions:—

A. At 0° C.
B. At the respective boiling-points under 1 atmosphere pressure.
C. At equally reduced temperatures (0·6396).
D. At the respective boiling-points under equally reduced pressures (0·02241).
E. At the respective critical-points.

The variation of the differences under the above conditions is thus undoubted. This confirms the older work by Gartenmeister on the ethereal salts of the fatty acids. In order to show that the apparent volume of CH_2 is not the true one the following considerations are submitted:—It is shown (*loc. cit.*) that there exists the relation $C = 4H$ between the volumes of the atoms C and H, so that it is merely necessary to divide the molecular volumes of the normal paraffins by an arithmetical series—similar to that

TABLE I.—*Volume of* CH_2 *in the Normal Paraffins under various Conditions (Young).*

Paraffin.	A. Mol. vol.	A. Δ.	B. Mol. vol.	B. Δ.	C. Mol. vol.	C. Δ.	D. Mol. vol.	D. Δ.	E. Mol. vol.	E. Δ.
n-Butane ..	—		96		—		—		251·0	
		15·44		21·80		20·09		20·86		58·3
n-Pentane ..	111·33		117·8		116·13		116·13		309·3	
		15·69		22·13		20·18		21·49		56·8
n-Hexane ..	126·77		139·93		136·22		137·19		366·1	
		15·88		22·63		20·54		21·83		60·2
n-Heptane ..	142·46		162·56		156·40		158·68		426·3	
				23·70						62·6
n-Octane ..	158·34		186·26		176·94		180·51		488·9	

TABLE II.—*The Volumes of* CH_2, *apparent and real, at the Boiling-point and at equally Reduced Pressure.*

Hydrocarbon.	W.	Mol. vol. at boiling-point.	Δ.	6 V/W.	Mol. vol. for P/P_k = 0·01179.	Δ.	6 V/W.
n-Butane, C_4H_{10} ..	26	96		22·15	—		—
			21·80			11·11	
n-Pentane, C_5H_{12} ..	32	117·8		22·08	116·13		21·77
			22·13			21·06	
n-Hexane, C_6H_{14} ..	38	139·93		22·09	137·19		21·66
			22·63			21·49	
n-Heptane, C_7H_{16} ..	44	162·56		22·17	158·68		21·64
			23·70			21·83	
n-Octane, C_8H_{18} ..	50	186·25		22·35	180·51		21·66

TABLE IV.—*Showing the Values of* V/W *at the Boiling- and the Critical-points.*

No.	Hydrocarbon.	W.	V (b. p.).	Δ.	V/W.	V *k*.	Δ.	V/W.
1.	*n*-Butane, C_4H_{10}	26	95·9		3·69	251·0		9·65
				21·9			58·3	
2.	*n*-Pentane, C_5H_{12}.. ..	32	117·8		3·68	309·3		9·67
				22·13			56·8	
3.	*n*-Hexane, C_6H_{14}	38	139·93		3·68	366·1		9·63
				22·63			60·2	
4.	*n*-Heptane, C_7H_{16} . ..	44	162·56		3·69	426·3		9·69
				23·70			62·5	
5.	*n*-Octane, C_8H_{18}	50	186·26		3·72	488·9		9·78
	Mean values				3·69			9·68

TABLE V.—*Volumes of the Normal Paraffin Hydrocarbons taken under Reduced Pressures.*

Reduced pressure.	Pentane, C_5H_{12}. W = 32. M.V.	V/W.	Hexane, C_6H_{14}. W = 38. M.V.	V/W.	Heptane, C_7H_{16}. W = 44. M.V.	V/W.	Octane, C_8H_{18}. W = 50. M.V.	V/W.
1·000000	309·20	9·66	366·30	9·64	425·7	9·67	488·9	9·77
0·82568	193·80	6·05	229·20	6·03	265·25	6·03	305·2	6·10
0·44232	153·85	4·808	181·80	4·784	210·36	4·781	241·11	4·822
0·144744	131·25	4·101	155·09	4·082	179·62	4·082	204·62	4·092
0·04232	120·15	3·755	141·95	3·736	164·02	3·728	187·02	3·740
0·022411	116·13	3·629	137·19	3·610	158·68	3·606	180·51	3·610
0·011795	113·20	3·537	133·65	3·517	154·58	3·513	175·83	3·517

of the valency numbers—to obtain the true volumes of H in each compound, and so the volumes of CH_2.

The assumption is, of course, made that all the atoms C and H obey the 4 : 1 rule in every individual compound. This is justified, for $H_2 = C_nH_{2n+2} - nCH_2 - \frac{1}{2}CH_2$, as will be seen. A comparison of the substances is made.

a. At the boiling-point under 1 atmosphere pressure.

b. Under identically reduced pressures P/P_k = 0·01179.

Since V/W represents the volume of H, W standing for the valency number of the compound, 6V/W represents the true volume of CH_2 in every individual compound.

Columns 4 and 7 give the true volumes of CH_2 under the above conditions, and it is seen that the molecular volumes are much more nearly additive than is shown by the apparent volumes of CH_2.

It must be remarked, however, that the changes from compound to compound are greater for more complex compounds than the above. A further consideration of this matter is reserved.

The above evidence, and also some to be afterwards mentioned, makes it clear that the limitations of the additive theory indicated by the variation of CH_2 is explained in the following way:—

III. *Although the volumes of like atoms are usually the same in a single compound they are yet susceptible to slight changes from compound to compound, but in such a way as to preserve the characteristic relationship between the volumes of dissimilar atoms.*

The above principle not only indicates the fact of variation of the molecular volume with the complexity of the compound, but also the manner of such variation.

A *fourth* principle shows the effect of a particular method of arrangement, namely, that of ring structure. It is as follows:—

IV. *The atomic volumes are smaller in a closed than in an open chain compound. The total diminutions which are characteristic of certain classes of compounds are functions not only of the number of atoms in a ring, but also of the number of rings.*

The method of finding these total diminutions is to deduct the molecular volume of the ring compound from the sum of the atomic volumes found from a consideration of open chain series. Another way of stating this is that the diminutions represent the differences between the volumes of the ring compounds and the volumes of similar complexes in the open chain series. From the fact that the diminutions are similar for the same class of compounds, it is concluded that the relative volumes of the atoms in ring compounds are similar to the relation between their volumes in the open chain series—a fact which further proves the validity of Principle III.

The following figures illustrate the importance of Generalisation IV., which shows the effect of ring structure on molecular volumes.

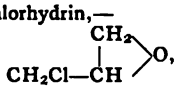
It will be necessary to account for smaller volume differences by slighter differences in arrangement, but this can only be done by a careful study of accurate data. As it has already been stated, one generalisation of importance has been given.

TABLE III.

	Contraction.
Three-membered single ring—	
Epichlorhydrin, C_3H_5ClO	−5·2
Four-membered single ring—	
Diamylene, $C_{10}H_{20}$..	−10·7
Five-membered ring—	
Thiophene, C_4H_4S	−11·5
Six-membered single ring—	
Paraldehyde, $C_6H_{12}O_3$	−15·8
Benzene, C_6H_6	−15·0
Carvene, $C_{10}H_{16}$	−16·9
Double ring—	
Pinene, $C_{10}H_{16}$	−25·7
Naphthalene, $C_{10}H_8$	−30·4
Camphor, $C_{10}H_{16}O$..	−30·6
Triple ring—	
Anthracene, $C_{14}H_{10}$	−48·7

A point of very great practical importance is that ring compounds, which are immediately derived from members of the open chain class of compounds like diamylene and paraldehyde—which are formed by the polymerisation of amylene and aldehyde respectively—are equally subject to contractions with such typical ring compounds as benzene and pyridene.

The case of epichlorhydrin,—



which is a very simple ring compound, shows the large diminution of −5·2; that is, its volume is smaller by 5·2 units than that of its theoretical isomer, $CHCl=CH—CH_2OH$. These facts show that we have here a valuable aid in the elucidation of chemical structure

It is thus clear that molecular volumes take into account two great types of structure, the open and closed chain, which types the theory of linking of atoms foreshadows, and which are completely identified by modern methods of chemical investigation.

It must be particularly observed that the above principles are not of the nature of explanations, so much as facts which require explanation. The nature of the latter must depend on the precise meaning to be attached to the term "atomic volume." This involves the far-reaching question of liquid structure about which very little is known.

The Atomic Volumes of C *and* H.—Kopp concluded from his observations that C = 11 and H = 5·5, so that C = 2H. Schröder's relation was C = H.

Neither of these relations are admissible (see *Phil. Mag.*, [6], No. 81, xiv., p. 338); Kopp's numbers were obtained by a comparison of the volumes of such differently constituted bodies as butane C_4H_{10}, and benzene C_6H_6, for example; and thus it was concluded that the very great differences in the constitutions of these compounds produce no effect on their molecular volumes. Since, however, molecular volumes are only in part additive, this would *à priori* appear improbable. It is far better to obtain the atomic volumes of C and H by comparing the volumes of related compounds, such as those belonging to the fatty series.

I. *The Volumes* C *and* H *in the Normal Saturated Paraffins.*

A point which is worthy of notice is that the molecular volumes of the normal paraffin hydrocarbons are proportional to their valency numbers at the boiling- and the critical-points, and indeed under all corresponding conditions, especially at identically reduced pressures (see *Phil. Mag.*, Sept., 1907, [6], No. 81, xiv., pp. 337—344). If V represents the molecular volume and W the valency number, then V/W is approximately constant under the above conditions.

The constancy of V/W is in each case far greater than the differences for CH_2 would lead us to expect, especially at the critical temperature. Since the latter are necessarily subject to perturbations which more or less mask the true value of CH_2, it is better to obtain it from the value of V/W, which represents the volume of H. Thus, $6 \times 3{\cdot}69 = 22{\cdot}1$ (approx.).

Now,—

$$C_nH_{2n+2} = nCH_2 + H_2,$$

and therefore,—

$H_2 = C_nH_{2n+2} - nCH_2$

$H_2 = (C_4H_{10}) - 4(CH_2) = 95{\cdot}9 - 88{\cdot}4 = 7{\cdot}5$

$= (C_5H_{12}) - 5(CH_2) = 117{\cdot}8 - 110{\cdot}5 = 7{\cdot}3$

$= (C_6H_{14}) - 6(CH_2) = 139{\cdot}93 - 132{\cdot}6 = 7{\cdot}33$

Mean value of $H_2 = 7{\cdot}38$

The direct determination of the volume of H at the boiling-point is—

$$H = \frac{7{\cdot}38}{2} = 3{\cdot}69,$$

and since $CH_2 = 22{\cdot}1$, therefore $C = 22{\cdot}1 - 7{\cdot}38 = 14{\cdot}72 = 4 \times 3{\cdot}68$.

The relation C = 4H is thus the true one. At the critical-point the true value of CH_2 is $6 \times 9{\cdot}68 = 58$, so that—

$H_2 = 251 - 23{\cdot}2 = 19$ for C_4H_{10}

$= 309{\cdot}3 - 290 = 19{\cdot}3$ for C_5H_{12}

$= 366{\cdot}1 - 348 = 18{\cdot}1$ for C_6H_{14}

$= 426{\cdot}3 - 406 = 20{\cdot}3$ for C_7H_{16}

Mean value of $H_2 = 19{\cdot}2$ (approx).

The volume of H at the critical-point is thus approx.,—

$$\frac{19{\cdot}2}{2} = 9{\cdot}6,$$

and since $CH_2 = 58$, $C = 58 - 19{\cdot}2 = 38\,8 = 4 \times 9{\cdot}7$.

The relation C = 4H is also true at the critical-point.

The difference for CH_2 is about the same in all the fatty series. It is thus concluded that carbon and hydrogen occupy similar volumes in the saturated hydrocarbon residues that they do in the normal paraffin hydrocarbon series.

It has been shown that the relation C = 4H holds at the melting-point and at equal intervals of temperature therefrom in the complex hydrocarbons from undecane $C_{11}H_{23}$, to pentatriacontane $C_{35}H_{72}$. Here, however, V/W, that is to say, H = 2·970 exactly, and $C = 4 \times 2{\cdot}970 = 11{\cdot}88$ (*Phil. Mag.*, Sept., 1907, No. 81, pp. 324—334).

The relation C = 4H is also true under conditions of equally reduced pressures, in the case of the simple normal paraffins, from pentane to octane (Table V.). (For data see Young, *Journ. Chem. Soc.*, 1895—1900).

The Unsaturated Hydrocarbons.—It is remarkable that the volumes of the olefines and the acetylenes are also proportional to their valency numbers, from which it follows that the relation C = 4H holds. The atomic volumes of C and H are C = 14·8 and H = 3·7. This furnishes a means of verifying the above numbers in a direct way (see Table VI.).

TABLE VI.—*The Volumes of the Atoms* C *and* H *in Unsaturated Compounds at the Boiling-point.*

Compounds.	W.	V.	Δ for $n(H_2)$.	V/W.	Authority.
Pentane, C_5H_{12}..	32	117·8		3·68	Young
Amylene, C_5H_{10}..	30	109·9	$2 \times 3{\cdot}95$	3·67	Schiff
Valerylene, C_5H_8.	28	104·5	$4 \times 3{\cdot}32$	3·73	Buff
Hexane, C_6H_{14}..	38	139·93		3·68	Young
Hexylene, C_6H_{12}.	36	132·4	$2 \times 3{\cdot}70$	3·68	Schiff
Diallyl, C_6H_{10} ..	34	125·8	$4 \times 3{\cdot}53$	3·71	Schiff
Heptane, C_7H_{16}..	44	162·56		3·69	Young
Heptylene, C_7H_{14}	42	154·58	$2 \times 3{\cdot}88$	3·68	Schiff
Octylene, C_8H_{16} .	48	177·6		3·70	Young
Mean values ..			$14 \times 3{\cdot}58$	3·69	

The value of V/W is found to be approximately constant in the unsaturated compounds, and the approximate value of H is shown by the differences, although the latter are necessarily somewhat irregular, being small numbers.

It nevertheless follows that V/W = H.

If the unsaturated members of other fatty series are also considered, the same differences for H_2 appear, so that additive relations are also found to be true for such compounds (Table VII.).

TABLE VII.—*The Volume of* H *in the various Fatty Series.*

P compounds.	V.	Δ.	Authority.
Propyl alcohol, C_3H_8O ..	81·3	2×3·55	Schiff
Allyl alcohol, C_3H_6O . ..	74·2		,,
Propyl amine, C_3H_9N ..	85·6	2×3·60	,,
Allyl amine, C_3H_5N.. ..	78·4		,,
Tripropyl amine, $N(C_3H_7)_3$	222·1	6×3·63	,,
Triallyl amine, $N(C_3H_5)_3$.	200·3		,,
Propyl acetate, $C_5H_{10}O_2$..	128·5	2×3·60	,,
Allyl acetate, $C_5H_8O_2$ ..	121·6		,,
Propyl chloride, C_3H_7Cl..	91·4	2×3·60	,,
Allyl chloride, C_3H_5Cl ..	84·2		,,
Mean value of H_2 ..		14×3·60	

The volume of H is shown by Table VII. to be the same in various fatty series as in the open chain hydrocarbons, saturated and unsaturated.

A similar direct method to the above of calculating the value of C is available (see Table VIII.).

TABLE VIII.—*The Volume of* C *in the "Straight Chain" Hydrocarbons.*

Hydrocarbon.	V.	Δ for C.
Butane, C_4H_{10}	95·9	14·0
Amylene, C_5H_{10}.. ..	109·9	16·0
Diallyl, C_6H_{10}	126·1	
Pentane, C_5H_{12}	117·8	14·6
Hexylene, C_6H_{12} . ..	132·4	
Hexane, C_6H_{14}	139·93	14·87
Hexylene, C_7H_{14} . ..	154·8	
Heptane, C_7H_{16}.. ..	162·56	15·04
Octylene, C_8H_{16}.. ..	177·6	
Mean value of C ..		14·9

Since the above numbers for C and H approximately agree with those found by considering the normal paraffins at the boiling-point, it does not appear that unsaturation has any special effect on the molecular volumes under the above conditions.

The conclusion is that at the boiling-point the volumes of carbon and hydrogen in the aliphatic class of compounds are C = 14·8, H = 3·7, *subject to augmentations as complexity increases, and that under corresponding conditions there exists the relation* C = 4H *between the atomic volumes* C *and* H.

The molecular volumes of the hydrocarbons at the boiling-point can thus be calculated by means of the following equations:—

Mol. vol of—

Normal paraffins $C_nH_{2n+2} = (6n+2)S = WS$
Olefines .. .. $C_nH_{2n} = (6nS) = WS$ $S = 3·70$
Acetylenes .. $C_nH_{2n-2} = (6n-2)S = WS$

(W represents the valency numbers).

The above atomic values of C and H are quite different from those of Kopp, C = 11 and H = 5·5. This change in the fundamental atomic values entails very important consequences in the molecular volume theory.

(To be continued).

THE

RELATION BETWEEN THE CRYSTALLINE FORM AND THE CHEMICAL CONSTITUTION OF THE PICRYL DERIVATIVES.*

By GEORGE JERUSALEM and WILLIAM JACKSON POPE, F.R.S., Professor of Chemistry, Municipal School of Technology, Victoria University of Manchester.

THE general relation existing between the constitution and the crystalline form of chemical substances has been determined by Barlow and Pope (*Trans. Chem. Soc.*, 1906, lxxxix., 1675; 1907, xci., 1150). It is conveniently illustrated by assigning to each atom present a sphere or domain of influence, and constructing homogeneous close-packed assemblages corresponding in composition and constitution to the compounds concerned, using spheres as structural units to represent the spheres of atomic influence. On constructing such assemblages it is found that, for their dimensions to accord with the geometrical dimensions of the crystal, the spheres of influence, delimited by the polyhedral cells enclosed by tangent planes drawn through the points of contact of neighbouring spheres, must possess volumes approximately proportional to the fundamental valencies of the corresponding elements. The assemblages derived in this way exhibit properties corresponding with the chemical composition and constitution, and with the crystalline form of the substance represented; they are found also to be homogeneously partitionable into units which correspond in configuration with the chemical properties of the substance.

The method of treatment here briefly indicated has been applied and tested to such an extent as proves its general applicability, and demonstrates that the principle involved is the correct one (Jaeger, *Trans. Chem. Soc.*, 1908, xciii., 517); the conclusion that the fractions of the molecular volume appropriated by the component atoms are approximately proportional to the valencies of the respective elements has also been substantiated by independent work on the subject of molecular volumes (Le Bas, *Trans. Chem. Soc.*, 1907, xci., 112; *Phil. Mag.*, 1907, xiv., 324).

As the theory indicates that the crystalline forms exhibited by related substances are derivable in a comparatively simple manner one from the other, it is desirable that groups of allied organic substances should be examined, as far as possible, by aid of the new method, We have therefore studied the whole of the crystallographic data available for the derivatives of the symmetrical radicle, picryl, namely, 1 : 3 : 5-trinitrophenyl, $C_6H_2(NO_2)_3$, and for purposes of comparison have determined experimentally the geometrical constants of several related substances.

Two distinct assemblages can be devised, both in accordance with the crystallographic and chemical data, to represent the crystal structure of benzene and its simple derivatives (*Trans. Chem. Soc.*, 1906, lxxxix., 1692); it has been shown that both these forms of assemblage can be traced among the simple derivatives of benzene. The first assemblage is derivable from the closest-packed assemblage of equal spheres of hexagonal type (*loc. cit.*, p. 1699), and, taking the volume of a monovalent sphere of atomic influence as unity, the dimensions of a unit of the partitioning of the assemblage, namely, of the benzene molecule itself, are determined by the following rectangular co-ordinates:—

$$x : y : z = 3·101 : 3·480 : 2·780.$$

These values constitute the so-called equivalence parameters for benzene and their product; the valency volume, W = 30, is the sum of the valencies of the atoms composing the benzene molecule, C_6H_6.

The second form of assemblage met with among the simple derivatives of benzene is derived from the closest-packed assemblage of equal spheres of cubic type, and is therefore of rhombohedral marshalling; it differs dimen-

* A Paper read before the Royal Society, April 30, 1908.

TABLE I.—*Crystal System (Orthorhombic).*

$a : b : c = 1{\cdot}8914 : 1 : 1{\cdot}3871$.

Forms observed: $a\{100\}$, $c\{001\}$, $r\{201\}$, $q\{021\}$, and $o\{111\}$.

Angle.	Number of observations.	Limits.	Mean.	Calculated.
001 : 201	15	55° 33′ to 55° 56′	55° 42′ 50″	—
100 : 111	12	66 34 „ 66 53	66 47 10	—
001 : 111	20	57 21 „ 57 58	57 32 20	57° 29′ 30″
111 : 1$\bar{1}$1	8	64 49 „ 65 17	64 59 0	65 1 0
201 : 20$\bar{1}$	5	68 21 „ 68 58	68 35 40	68 34 10
201 : 111	9	50 56 „ 51 20	51 5 30	51 3 50
111 : 021	4	27 45 „ 27 51	27 47 0	27 55 10
001 : 021	2	70 17 „ 70 22	70 19 30	70 10 40

TABLE II.—*Crystalline System (Hexagonal).*

$a : c = 1 : 1{\cdot}3890$.

Forms observed: $\{10\bar{1}1\}$, $\{10\bar{1}0\}$, $\{11\bar{2}0\}$, and $\{0001\}$.

Angle.	Number of observations.	Limits.	Mean.	Calculated.
10$\bar{1}$1 : 01$\bar{1}$1	8	50° 17′ to 50° 3′	50° 12′ 40″	—
0001 : 01$\bar{1}$1	15	57 45 „ 58 25	58 2 50	58° 3′ 20″
11$\bar{2}$0 : 01$\bar{1}$1	6	42 37 „ 43 5	42 43 30	42 42 10
10$\bar{1}$0 : 10$\bar{1}$1	5	31 42 „ 32 5	31 57 0	31 56 40

TABLE III.

Substance.	$x : y : z$	β.	W.	Crystal system.
Substances of hexagonal marshalling—				
C_6H_6 ($y : z : x$)	3·480 : 2·780 : 3·101	90°	30	Orthorhombic.
1 : 3 : 5-$C_6H_2(NO_2)_3ONH_4$	5·251 : 2·776 : 3·852	90°	56	„
1 : 3 : 5-$C_6H_2(NO_2)_3OK$	5·082 : 2·699 : 3·647	90°	50	„
1 : 3 : 5-$C_6H_2(NO_2)_3CO_2H$.. ..	4·816 : 2·753 : 4·334	90°	56	„
1 : 3 : 5-$C_6H_2(NO_2)_3OCH_3$.. ..	4·843 : 2·811 : 4·115	89° 1′	56	Monosymmetric.
1 : 3 : 5-$C_6H_2(NO_2)_3NH_2$	4·815 : 2·873 : 3·814	80° 11′	52	„
1 : 3 : 5-$C_6H_2(NO_2)_3CH_3$	5·206 : 2·777 : 3·735	90°	54	Orthorhombic.
1 : 3 : 5-$C_6H_2(NO_2)_3Cl$	5·013 : 2·804 : 3·414	89° 56′	48	Monosymmetric.
1 : 3 : 5-$C_6H(NO_2)_3(OH)_2$	4·825 : 2·786 : 3·869	90°	52	Hexagonal.
1 : 3 : 5-$(NO_2)_3C_6(OH)_3.H_2O$ ..	4·822 : 2·784 : 4·320	90°	58	„
Substances of rhombohedral marshalling—				
1 : 3 : 5-$C_6H_3(NO_2)_3$	4·921 : 2·579 : 3·781	90°	48	Orthorhombic.
1 : 3 : 5-$C_6Br_3(NO_2)_2CH_3$	4·909 : 2·561 : 3·831	α=85° 12′	48	Monosymmetric.
1 : 3 : 5-$C_6H_2(NO_2)_3OH$	3·706 : 3·596 : 3·572	90°	50	Orthorhombic.
1 : 3 : 5-$C_6H_2(NO_2)_3I$	3·660 : 3·583 : 3·660	90°	48	Tetragonal.

sionally from the hexagonal type by exhibiting a smaller z value and a larger y value than the former.

From the papers referred to it appears that among the simple derivatives of benzene the dimension z varies less than do the other two, namely, x and y; it would therefore seem that the adjustment of the assemblage which restores close-packing after the introduction of a substituting group into the benzene structure in general exerts itself mainly in the directions of x and y, and only affects but slightly the dimension z. The direction of z is that of the vertical dimension in the benzene molecule; that is to say, of the dimension perpendicular to the two planes containing the centres of the two sets of hydrogen atoms ordinarily numbered 1 : 3 : 5 and 2 : 4 : 6, respectively. It will be shown below that this approximate constancy of the vertical or z dimension is maintained in the more complex picryl derivatives.

Ammonium Picrate, C_6H_2—1 : 3 : 5—$(NO_2)_3$—ONH_4.

Ammonium picrate crystallises from water in orthorhombic forms; these have been measured by Handl (*Ber. k. Akad. Wien*, 1858, xxxii., 259) and by Laurent (*Revue Scient.*, ix., 26), who respectively assign to the substance the axial ratios:—

$$a : b : c = 1 : 0{\cdot}6787 : 0{\cdot}3569$$

and—

$$a : b : c = 1 : 0{\cdot}6873 : 0{\cdot}3652,$$

the mean values being—

$$a : b : c = 1 : 0{\cdot}6830 : 0{\cdot}3611.$$

Crystals much more suitable for exact measurement than these are obtained by crystallising the salt from acetone, in which it is fairly soluble; on allowing the acetone solution to evaporate spontaneously at the ordinary temperature, large transparent yellow crystals of hexagonal habit are deposited. The bright yellow colour of the crystals thus produced is quite distinct from the red tint sometimes assumed by ammonium picrate (Silberrad, *Trans. Chem. Soc.*, 1908, xciii., 477). The forms $a\{100\}$ and $c\{001\}$ are generally the best developed, and $q\{021\}$ was only observed on one crystal; the results obtained on measurement are given in Table I.

The form $o\{111\}$ has not previously been observed on

ammonium picrate crystals. The crystals exhibit a perfect cleavage on $a\{100\}$; this is also the optic axial plane, and the axis b is the acute bisectrix. The optic axial dispersion is very marked; the angle for red is much larger than that for yellow light.

The crystals deposited from water and from acetone are evidently of identical structure, for, on writing the axial ratios now given, namely,—

$$a : b : c = 1{\cdot}8914 : 1 : 1{\cdot}3871,$$

in the form—

$$a' : b' : c' = 1 : a/2c : 1/2c = 1 : 0{\cdot}6818 : 0{\cdot}3609,$$

he result is practically identical with the mean of the measurements of Handl and Laurent, namely,—

$$a : b : c = 1 : 0{\cdot}6830 : 0{\cdot}3611.$$

On calculating the equivalence parameters of ammonium picrate from our determination of the axial ratios, taking the valency volume of the molecule, $C_6H_2(NO_2)_3ONH_4$, as W = 56, the following values are obtained :—

$$x : y : z = 5{\cdot}251 : 2{\cdot}776 : 3{\cdot}582.$$

For comparison with these equivalence parameters it is convenient to calculate those of potassium picrate; this salt is orthorhombic with—

$$a : b : c = 1{\cdot}4352 : 1 : 1{\cdot}0616$$

(Brugnatelli, *Zeit. Kryst. Min.*, 1895, xxiv., 277), and by transposition the axial ratios may be obtained in the form—

$$a : b : c = 2 : 1{\cdot}0616 : 1{\cdot}4352 = 1{\cdot}8839 : 1 : 1{\cdot}3519.$$

The latter values show clearly the isomorphism of this salt with the previous one; the equivalence parameters of potassium picrate, $C_6H_2(NO_2)_3OK$, with W = 50 are calculated from the last form of the axial ratios as—

$$x : y : z = 5{\cdot}082 : 2{\cdot}699 : 3{\cdot}647.$$

It is seen that on passing from the potassium to the ammonium salt the principal increase amongst the equivalence parameters occurs in the directions x and z, whilst the value of y changes to a smaller extent, namely, from 2·699 to 2·776. The dimension y in the cases of these salts obviously corresponds to the dimension $z = 2{\cdot}780$ in benzene itself, and this dimension, as remarked above, is the one of the three which tends to change least on simple substitution.

Ammonium picrate has the same valency volume, W = 56, as the orthorhombic 1 : 3 : 5-trinitrobenzoic acid, $C_6H_2(NO_2)_3CO.OH$, with the axial ratios—

$$a : b : c = 0{\cdot}8770 : 1 : 0{\cdot}5715$$

(Friedländer, *Zeit. Kryst. Min.*, 1877, i., 623), on interchanging in these axial ratios, c and b, b and a, and a and c, these values become—

$$a : b : c = 1 : 0{\cdot}5715 : 0{\cdot}8770 = 1{\cdot}7497 : 1 : 1{\cdot}5346.$$

This set of axial ratios corresponds to the equivalence parameters—

$$x : y : z = 4{\cdot}816 : 2{\cdot}753 : 4{\cdot}224.$$

The y value again corresponds closely to that of ammonium picrate and to the z value of benzene, so that on replacing the group, ONH_4, in the salt by the group, CO_2H of the same valency volume, W = 9, the vertical benzene dimension is sensibly preserved.

The methyl ether of picric acid 1 : 3 : 5 - trinitroanisol, $C_6H_2(NO_2)_3OCH_3$, also has the same constitution and valency volume, W = 56, as ammonium picrate; it crystallises in the monosymmetric system (Friedländer, *Zeit. Kryst. Min.*, 1879, iii., 173; compare Jaeger, *Ibid.*, 1905, xl., 565) with the axial ratios—

$$a : b : c = 1{\cdot}4635 : 1 : 2{\cdot}2211, \beta = 50° 48'.$$

These axial ratios may be transposed by assigning to the planes (100), (10$\bar{1}$), and (110) the indices (10$\bar{1}$), (001), and (11$\bar{1}$), respectively, and then become—

$$a : b : c = 1{\cdot}7225 : 1 : 1{\cdot}4635, \beta = 91° 51'.$$

The equivalence parameters corresponding to these axia ratios are—

$$x : y : z = 4{\cdot}843 : 2{\cdot}811 : 4{\cdot}115, \beta = 91° 59' \text{ or } 89° 1'.$$

The y value is again approximately equal to the z value for benzene and the substitution of the methoxyl group, $.OCH_3$, for $.ONH_4$ or $.CO_2H$, has effected but little change in the dimensions of the crystal structure.

Picramide, $C_6H_2(NO_2)_3NH_2$, which may be regarded as derived from ammonium picrate by the elimination of one molecule of water, crystallises in the monosymmetric system with (Friedländer, *loc. cit.*)—

$$a : b : c = 0{\cdot}838 : 1 : 0{\cdot}6638, \beta = 80° 11°.$$

After dividing the length b by two, so that the axial ratios become—

$$a : b : c = 1{\cdot}676 : 1 : 1{\cdot}3276, \beta = 80° 11',$$

and calculating the equivalence parameters, taking the valency volume as W = 52, the following values are obtained :—

$$x : y : z = 4{\cdot}815 : 2{\cdot}873 : 3{\cdot}814, \beta = 80° 11'.$$

The y value differs rather more than before from the z value for benzene, but the z value is almost the same as for ammonium picrate.

The symmetrical trinitrotoluene,—

$$C_6H_2\text{-}1 : 3 : 5\text{-}(NO_2)_3\text{-}6\text{-}CH_3,$$

has a valency volume two units less than that of ammonium picrate and trinitroanisol, namely, W = 54, and crystallises in the orthorhombic system with—

$$a : b : c = 0{\cdot}9373 : 1 : 0{\cdot}6724$$

(Friedländer, *loc. cit.*). On dividing the length b by two, as in the previous case, so as to obtain the axial ratios in the form—

$$a : b : c = 1{\cdot}8746 : 1 : 1{\cdot}3448,$$

and calculating the equivalence parameters, the following values are obtained :—

$$x : y : z = 5{\cdot}206 : 2{\cdot}777 : 3{\cdot}735.$$

In this case both the x and y values are nearly identical with those for ammonium picrate, and the effect of the change of valency volume is thrown, in the main, upon the direction z.

Picryl chloride, $C_6H_2Cl\text{-}1 : 3 : 5\text{-}(NO_2)_3$, derived from picric acid by substituting chlorine for hydroxyl, has the valency volume, W = 48, and crystallises in the monosymmetric system with—

$$a : b : c = 1{\cdot}83288 : 1 : 0{\cdot}81135, \beta = 77° 9'$$

(Bodewig, *Zeit. Kryst. Min.*, 1879, iii., 398). On transposing these axial ratios so that the planes (001), (10$\bar{1}$), (101), and (110) become respectively (103), (10$\bar{1}$), (30$\bar{1}$), and (110), the following values result :—

$$a : b : c = 1{\cdot}7875 : 1 : 1{\cdot}2175, \beta = 90° 4'.$$

The corresponding equivalence parameters are—

$$x : y : z = 5{\cdot}013 : 2{\cdot}804 : 3{\cdot}414, \beta = 90° 4' \text{ or } 89° 56'.$$

The y dimension in this case also retains approximately its previous value.

Styphnic acid, $C_6H(OH)_2\text{-}1 : 3 : 5\text{-}(NO_2)_3$, the monohydroxy-derivative of picric acid, separates on spontaneous evaporation of its alcoholic solution in large straw-yellow prisms belonging to the hexagonal system; the results obtained on measurement are given in Table II.

The first angle quoted, namely, 50° 12′ 40″, has been given by Ditscheiner as 50° 12′ (*Annalen*, 1871, clviii., 247). On examining a section cut perpendicular to the principal axis, the uniaxial interference figure of the normal type is seen; the double refraction is strong and negative in sign. No indications were obtained of the hemimorphous development described by Lehmann (*Zeit. Kryst. Min.*, 1882, vi., 51).

For the purpose of comparing this substance with those

discussed above, its crystal form is conveniently referred to an orthorhombic axial system, in which the new ratio,—

$$a' : b' : c' = a\sqrt{3} : a : c = 1{\cdot}7321 : 1 : 1{\cdot}3890.$$

The equivalence parameters of styphnic acid, for which W = 52, are thence calculated as—

$$x : y : z = 4{\cdot}825 : 2{\cdot}786 : 3{\cdot}869.$$

The y value is again practically identical with the z value for benzene.

The symmetrical trinitrophloroglucinol crystallises with water, the crystals having the composition $C_6(OH)_3$-1 : 3 : 5-$(NO_2)_3,H_2O$, and belonging to the hexagonal system with—

$$a : c = 1 : 1{\cdot}0346$$

(Ditscheiner, *Zeit. Kryst. Min.*, 1881, v., 646). On increasing the length c by one-half and referring the axial ratios to an orthorhombic system of axes just as in the previous case, the axial ratios—

$$a : b : c = 1{\cdot}7231 : 1 : 1{\cdot}5519$$

are obtained. The equivalence parameters calculated from these values, taking W = 58, are—

$$x : y : z = 4{\cdot}822 : 2{\cdot}784 : 4{\cdot}320.$$

The values for x and y are practically identical with those for styphnic acid, so that the morphotropic effect of introducing the group, OH,H_2O, in place of a hydrogen atom in the styphnic acid assemblage is merely to increase the dimension z. Cases of very similar character to this occur among the minerals of the humite series and between camphoric anhydride and the addition compound of camphoric acid with acetone (*Trans. Chem. Soc.*, 1906, lxxxix., 1685).

The above results show that, by applying the method given by Barlow and Pope to the crystallographic data, an extensive series of picryl derivatives can be at once referred in a very simple manner to the hexagonal assemblage described for benzene. The following picryl derivatives seem to be derived from the alternative benzene assemblage, that, namely, which possesses the rhombohedral type of marshalling.

The symmetrical trinitrobenzene, 1 : 3 : 5-$C_6H_3(NO_2)_3$, is orthorhombic with the axial ratios (Friedländer, *loc. cit.*, p. 170)—

$$a : b : c = 0{\cdot}9540 : 1 : 0{\cdot}7330.$$

On dividing the length b by two, so as to obtain the axial ratios—

$$a : b : c = 1{\cdot}9080 : 1 : 1{\cdot}4660,$$

and calculating the equivalence parameters, with W = 48, the following values result :—

$$x : y : z = 4{\cdot}921 : 2{\cdot}579 : 3{\cdot}781.$$

The y value is in this case much smaller than among the previously discussed substances.

The 1 : 3 : 5-trinitrobenzene is identical in constitution and valency volume with the tribromo-1 : 3-dinitro-5-methylbenzene, and the two substances are morphotropically very closely related ; the latter is monosymmetric with—

$$a : b : c = 0{\cdot}5217 : 1 : 0{\cdot}7803, \beta = 85° 12'.$$

(Jaeger, *Zeit. Kryst. Min.*, 1905, xl., 360). On interchanging the axial directions a and b, the axial ratios are obtained in the form—

$$a : b : c = 1{\cdot}9168 : 1 : 1{\cdot}4957, \alpha = 85° 12'.$$

These values correspond to the equivalence parameters—

$$x : y : z = 4{\cdot}909 : 2{\cdot}561 : 3{\cdot}831, \alpha = 85° 12'.$$

The above numbers show that in the passage from the symmetrical trinitrobenzene to the dinitrotribromotoluene of similar constitution, which results from replacing one nitro-group in the former by a methyl-group of the same valency volume, and replacing three hydrogen atoms by three bromine atoms, also of the same valency, practically no change is effected in the relative dimensions; the equivalence parameters are scarcely affected by the substitution.

It has been already remarked that the differences in dimensions between the benzene assemblages of hexagonal and of rhombohedral marshalling are greatest in the directions of y and z, so that the dimension x remains nearly the same in both. A similar correspondence would be expected to hold among the derivatives of benzene, and accordingly it is found that the x values, 4·909 and 4·921, of the dinitrotribromotoluene and the trinitrobenzene respectively, are nearly identical with the x value, 5·013, of picryl chloride, the three substances having the same valency volume.

These correspondences strongly indicate that the above two substances exhibit in their crystalline structure the second form of the benzene assemblage ; the same conclusion presumably holds for picric acid and picryl iodide, both of which crystallise in pseudo-cubic forms.

Picric acid crystallises in the orthorhombic system with—

$$a : b : c = 1{\cdot}0305 : 1 : 1{\cdot}0434$$

(Brugnatelli, *Zeit. Kryst. Min.*, 1895, xxiv., 277), and has the valency volume W = 50. The equivalence parameters are—

$$x : y : z = 3{\cdot}706 : 3{\cdot}596 : 3{\cdot}752.$$

Picryl iodide, C_6H_2I-1 : 3 : 5-$(NO_2)_3$, with W = 48, crystallises in the tetragonal system with—

$$a : c = 1 : 1{\cdot}958$$

(Fels, *Zeit. Kryst. Min.*, 1900, xxxii., 364). On halving the length c, and writing the axial ratios in orthorhombic form, the equivalence parameters are obtained as—

$$x : y : z = 3{\cdot}660 : 3{\cdot}583 : 3{\cdot}660.$$

These values approximate very closely to those obtained for picric acid, and indicate that the replacement of iodine in picryl iodide by hydroxyl, to yield picric acid, results in the corresponding assemblage being slightly expanded in all directions, in accordance with the second geometrical property described by Barlow and Pope (*Trans. Chem. Soc.*, 1907, xci., 1204).

The equivalence parameters determined above are collected in Table III.

An inspection of the equivalence parameters of those picryl derivatives which assume the hexagonal type of marshalling shows, as previously noted, that the substitution in the benzene assemblage occurs so that groups which enter the benzene assemblage as substituting radicles are accommodated as the result mainly of an expansion of the assemblage in the directions of x and y ; the z dimension of benzene can thus be distinctly traced throughout the series of picryl derivatives as the y dimension of the latter. This conclusion indicates that the columns, consisting of superposed layers of three triangularly arranged carbon spheres, which form the skeleton of the crystalline benzene assemblage (*Trans. Chem. Soc.*, 1906, lxxxix., 1695), exist in all these crystalline substances, and that the corresponding assemblages are derived by pushing these columns apart, and packing the substituting groups into the space thus provided. The equivalence parameters now given furnish the data requisite to the construction of diagrams, similar to those previously given by Barlow and Pope, which show the precise mutual arrangement of the constituents of the substituting radicles in the picryl derivatives.

If the conclusion drawn concerning the existence of the columns of carbon spheres in these assemblages is of general application, it should be possible to detect the z dimension for benzene throughout all the benzene derivatives of hexagonal marshalling.

No difficulty appears to exist in applying the method of treatment used in this paper to the more complex picryl derivatives. Thus, acetamide picrate, $CH_3.CO.NH_2,C_6H_2(NO_2)_3OH$, crystallises in the orthorhombic system with—

$$a : b : c = 0{\cdot}4849 : 1 : 0{\cdot}3544$$

(Wyrouboff, *Ann. Chim. Phys.*, 1895, [7], v., 99). The valency volume, W = 68, and the corresponding equivalence parameters are—

$$x : y : z = 3{\cdot}560 : 7{\cdot}342 : 2{\cdot}602.$$

Further, isoapiol picrate, $C_{12}H_{14}O_4,C_6H_2(NO_2)_3OH$, crystallises in the monosymmetric system with—

$$a : b : c = 0{\cdot}9163 : 1 : 0{\cdot}4226, \beta = 89° 55'$$

(Boeris, *Zeit. Kryst. Min.*, 1905, xl., 106). The valency volume, W = 120, and the corresponding equivalence parameters are—

$$x : y : z = 6{\cdot}201 : 6{\cdot}767 : 2{\cdot}860, \beta = 89° 55'.$$

In each of these cases one of the three equivalence parameters has a value not greatly different from the value, $z = 2{\cdot}780$, for benzene: this would be expected if the columns of carbon spheres present in the benzene assemblage remain intact in these compounds.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

Part III.—Analysis of the Aluminium and Iron Groups, including Beryllium, Uranium, Vanadium, Titanium, Zirconium, and Thallium.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 83).

**Procedure* 58*b*. — To the $NaHCO_3$ filtrate add HNO_3 (1·20) until the solution is distinctly acid, avoiding an excess of more than 1 cc. (Colourless solution, absence of *chromium*). Unless the solution is as colourless as pure water, test one-fourth of it for chromium by adding to the cold solution in a test-tube 2 cc. ether and 0·5—1·0 cc. 3 per cent H_2O_2 solution. (Blue coloration of the ether layer, presence of *chromium*). To this mixture add about 5 cc. HNO_3 (1·42). Red coloration of water layer, presence of *vanadium*).

If chromium is not present, treat the HNO_3 solution or the part of it not tested with H_2O_2 by *P. 58*c*.

If chromium is present, exactly neutralise the remainder of the HNO_3 solution with NaOH, add 2 cc. HNO_3 (1·20) and then 20 cc. 20 per cent $Pb(NO_3)_2$ solution; allow the mixture to stand for fifteen to twenty minutes, and filter. (Yellow precipitate, presence of *chromium*). Saturate the filtrate with H_2S, filter off and reject the precipitate; boil the filtrate for two or three minutes to expel H_2S and to coagulate any sulphur that may separate; filter off and reject the precipitate. In order to oxidise vanadyl salts to vanadate, add bromine water (or if much is required, liquid bromine) until the solution has a permanent reddish colour. Boil in a casserole until the bromine is expelled and treat the solution by *P. 58*c*.

Notes.

1. The $NaHCO_3$ filtrate should not be heated after acidifying because some H_2O_2 may still be present (even though most of it is decomposed in the heating with $NaHCO_3$), which would cause the reduction of chromate, and might thus prevent the detection of even several milligrms. of chromium. (See *P. 58*a*, N. 2).

2. It is desirable to determine in advance, by making a preliminary test if necessary, whether or not chromium is present, for, if it is absent, the addition of the lead salt and the subsequent removal of the lead with H_2S may be omitted. If lead be added, its removal is necessary before the tests for uranium and vanadium can be made. If the acid solution is perfectly colourless, an amount of chromium exceeding 0·5 mgrm. may safely be pronounced absent. Uranium and especially vanadium in moderate quantity (20 to 50 mgrms.) also give yellow solutions. A yellow solution is therefore tested for chromate with H_2O_2. In regard to this test, see P. 56, N. 1. The test can be made in only a small portion (one-fourth) of the solution, for it is so delicate that the presence of 0·1 mgrm. chromium in this portion can be detected. The portion in which the test is made is rejected, because chromium, if present, is reduced to the chromic state by the H_2O_2 in the acid solution, and would therefore not be precipitated by the lead nitrate.

3. When vanadium is present even in moderately small quantity, the water layer, on the addition of H_2O_2, becomes orange-yellow to orange-red in colour, owing to the formation of pervanadic acid. This test for vanadium becomes more delicate when the solution is made strongly acid as is directed; a distinct colour is then obtained even when only 0·5 mgrm. vanadium is present in the portion tested, corresponding to about 2 mgrms. in the whole solution. (Compare *P. 51*a*, N. 4). The test is not essential since vanadium is always tested for later; it is introduced here, where it can be made in a moment's time, as an additional confirmation of the presence or absence of that element.

4. The separation of chromium from vanadium and uranium by $Pb(NO_3)_2$ depends on the relatively small solubility of lead chromate in dilute HNO_3 in the presence of a large quantity of $Pb(NO_3)_2$. Under the conditions described in the procedure, over 100 mgrms. of vanadium yield no precipitate and only 0·1 to 0·3 mgrm. of chromium usually remains in solution, though this amount is somewhat increased when a very large quantity of $NaNO_3$ has been introduced. To secure these results, however, care must be taken to use the prescribed quantities of HNO_3 and $Pb(NO_3)_2$, for lead vanadate is also a difficultly soluble substance, and would be quantitatively precipitated in the presence of a much weaker acid, such as acetic acid in the presence of ammonium acetate. Lead uranate would also be precipitated from a neutral or slightly alkaline solution.

5. The presence of much chloride or sulphate would cause the precipitation of white $PbCl_2$ or $PbSO_4$, which might obscure the yellow colour of a small quantity of $PbCrO_4$ and prevent the estimation of the amount of chromium present. These anions will, however, not be present in harmful quantity, if in dissolving the original $(NH_4)_2S$ precipitate (P. 51) the acids are used in the way prescribed, and if in acidifying the solutions in *P. 58*a* and *b*, HNO_3 and not HCl is used, as directed.

6. The lead which is added to precipitate the chromate must be removed before testing for uranium with Na_2HPO_4 (in *P. 58*c*), since it would give a precipitate of $Pb_3(PO_4)_2$. It is precipitated with H_2S, rather than with $(NH_4)_2SO_4$, because in the latter case enough lead (about 1 mgrm.) still remains in solution to give a precipitate with Na_2HPO_4.

7. By the H_2S, vanadic acid is reduced to vanadyl nitrate $VO(NO_3)_2$ slowly in the cold but more rapidly on heating the solution, so that sulphur may be precipitated on boiling the filtrate to expel H_2S. This must be re-oxidised to vanadic acid, for otherwise vanadyl ammonium phosphate would be precipitated with the uranium phosphate in *P. 58*c*. The oxidation by bromine does not take place instantaneously; and a considerable excess of bromine must therefore be used, and the mixture allowed to stand for a few minutes.

**Procedure* 58*c*.—Make the solution obtained in *P. 58*b* neutral with NH_4OH, add 5 cc. 30 per cent acetic acid, 1—2 grms. solid $(NH_4)_2SO_4$ (or NH_4NO_3), and then 2 grms. solid $Na_2HPO_4.12H_2O$, and heat to boiling. (White precipitate, presence of *uranium*). Allow the mixture to stand ten to fifteen minutes to coagulate the precipitate, filter, and wash the precipitate with a 5—10 per cent solution of $(NH_4)_2SO_4$ (or NH_4NO_3). (Precipitate, *P. 58*d*; filtrate, *P. 58*e*).

* From the *Journal of the American Chemical Society*, xxx., No. 4.

Notes.

1. The precipitation of white uranyl ammonium phosphate, $UO_2NH_4PO_4$, is a delicate test provided the solution be made only moderately acid with acetic acid and a sufficient excess of Na_2HPO_4 be added. The separation from vanadium as vanadic acid is a very satisfactory one, for vanadium, even when present in large quantity (100 mgrms.), does not precipitate, nor is a small quantity carried down by uranium; on the other hand, 0·5 mgrm. uranium gives a distinct precipitate. The test is a somewhat more delicate one if made in a smaller volume, say 40 cc.; in this case the same quantities of reagents may still be used, and the separation is perfectly satisfactory.

2. The formation of a precipitate at this point is not sufficient evidence of the presence of uranium, for aluminium, beryllium, and lead will separate as phosphates if they have not been completely removed in previous procedures, and vanadium will precipitate as vanadyl ammonium phosphate if the oxidation by bromine was incomplete. On the other hand, a slight turbidity may correspond to an appreciable amount of uranium (0·2 to 0·5 mgrm.). Therefore the confirmatory test for uranium (*P. 58*d*) should always be tried.

3. When the solution is made alkaline with NH_4OH a pale yellow precipitate of uranyl ammonium vanadate may separate, and may not dissolve when the acetic acid is added. This, however, has no effect on the separation, since, on boiling, it is converted into the less soluble uranyl ammonium phosphate and vanadic acid.

4. In the absence of ammonium salts uranium is precipitated as uranyl hydrogen phosphate, UO_2HPO_4, which often separates as a very finely divided precipitate that runs through the filter. The ammonium salt is added to cause the uranium to precipitate as uranyl ammonium phosphate, which coagulates and filters more readily.

**Procedure* 58*d*. — Dissolve the Na_2HPO_4 precipitate (*P. 58*c*) by pouring a small portion of hot HCl (1·12) repeatedly through the filter, evaporate the solution nearly to dryness, add about 10 cc. nearly saturated NaCl solution, pour into a test-tube, cool, and add 5 cc. 10 per cent $K_4Fe(CN)_6$ solution. (Dark red precipitate or coloration, presence of *uranium*).

Notes.

1. The precipitation of the dark red uranyl ferrocyanide $(UO_2)_2Fe(CN)_6$ is a very characteristic and also a very delicate test for uranium, provided care be taken to avoid any excess of acid. The solution must, however, be distinctly acid, for otherwise the precipitate may not form, owing to the small concentration of the uranyl ion, $UO_2{+}{+}$. Uranyl ferrocyanide tends to form a colloidal solution; but the presence of NaCl and HCl soon causes it to coagulate.

Procedure 58*e*.—Neutralise the filtrate from the Na_2HPO_4 precipitate (*P. 58*c*) with NH_4OH (0·90) and add at least 5 cc. more. Saturate the solution completely with H_2S by passing the gas through it in a small flask for ten or fifteen minutes. (Pink or violet red colour, presence of *vanadium*). Pour the solution through a filter; make it distinctly acid with acetic acid (or HCl) and heat nearly to boiling. (Dark precipitate, presence of *vanadium*).

Notes.

1. In regard to the formation of the sulphovanadate of vanadium in the strongly alkaline solution, the partial precipitation of vanadium sulphide on acidifying, and the action of H_2O_2 on vanadic acid, see the Notes on *P. 51*a*.

Procedure 58*f*.—Heat the $NaHCO_3$ precipitate (*P. 58*a*) with 10—30 cc. HCl (1·06) in a casserole, or dissolve it by pouring the acid two or three times through the filter, boil the solution to expel CO_2, add NH_4OH (0·96) until the solution after shaking smells of it, and then 2—3 cc. in excess. Heat the mixture nearly to boiling, filter and wash the precipitate, using suction if it seems desirable. (Precipitate, *P. 58*g*; filtrate, acidify with acetic acid and test for zinc by P. 57).

Notes.

1. The zinc remains in the ammoniacal filtrate as a complex ammonia salt, chiefly $Zn(NH_3)_4{+}{+}Cl_2{-}$. Aluminium and beryllium are precipitated as hydroxides. The precipitate will also contain any uranyl vanadate that was precipitated in the $NaHCO_3$ separation.

**Procedure* 58*g*. — Dissolve the NH_4OH precipitate (*P. 58*f*) by pouring a hot 5—15 cc. portion of HCl (1·12) repeatedly through the filter, using another portion of acid, if necessary. Add a volume of ether equal to one and a-half times that of the solution. Pass into the mixture in a small flask HCl gas until a single layer results, and until fumes of HCl are copiously evolved, cooling the flask in running water during the progress. Cover the flask and let the mixture stand for fifteen minutes even if no precipitate has separated. (White crystalline precipitate, presence of *aluminium*). Filter through an asbestos filter (see P. 61) or an ordinary filter supported by a small hardened filter folded with it, after first moistening the filter with a mixture of two volumes of HCl (1·20) and three of ether previously saturated with HCl gas; wash the precipitate once with this mixture. During the filtration and washing keep the funnel covered with a watch-glass to prevent evaporation of the ether. (Precipitate, P. 54, to confirm presence of aluminium; filtrate, *P. 58*h*).

Notes.

1. Aluminium chloride, $AlCl_3.6H_2O$, is only slightly soluble in concentrated HCl solutions and the precipitate is complete when ether is added and the mixture saturated with HCl. 0·5 mgrm. of aluminium can be easily detected in 30 cc. The test is therefore a delicate one. It is also very characteristic; for no other element of the aluminium group is precipitated by this treatment, except chromium when it is present in moderate quantity.

2. The ethereal solution of HCl, unlike the concentrated aqueous solution, does not disintegrate filter-paper rapidly, and the filtration can almost always be made with an ordinary filter supported by means of a hardened filter. This filtration is apt to be slow, and it is often advantageous to filter through asbestos with the help of suction.

3. When the NH_4OH precipitate is small (corresponding to less than 30 mgrms. Al), it need not be treated by this procedure, but it may be dissolved in a little HCl, and treated directly by *P. 58*h*.

**Procedure* 58*h*.— Evaporate the filtrate from the HCl precipitate (*P. 58*g*), first on a water-bath under a hood until the ether is expelled, and then over a flame, almost to dryness until nearly all the HCl is expelled; add a little water, make alkaline with NH_4OH, avoiding a large excess, and heat nearly to boiling. (No precipitate, absence of *beryllium*).

If there is a precipitate, dilute to 30 cc., add enough solid $NaHCO_3$ to make a 10 per cent solution, heat to boiling, boil for one minute, cool, pass in H_2S for a few seconds, and filter after a few minutes if there is a precipitate. Acidify the filtrate with HCl, boil for two or three minutes to expel CO_2, make the solution alkaline with NH_4OH, and heat nearly to boiling. (White flocculent precipitate, presence of *beryllium*; yellow precipitate, presence of *uranium*).

Treat this precipitate or a portion of it corresponding to about 20 mgrms. of beryllium by *P. 58*d*. (Dark red precipitate, presence of *uranium*). Filter, and to the filtrate add NH_4OH. (White precipitate, presence of *beryllium*).

Notes.

1. The filtrate from the HCl precipitate may contain besides beryllium, uranium or vanadium (carried down in the treatment with dilute $NaHCO_3$ in *P. 58*a*), aluminium (if care was not taken in precipitating and filtering aluminium chloride), and a little iron (introduced from the reagents). This solution is first tested with NH_4OH to determine if any further treatment is necessary. The treatment with the hot concentrated $NaHCO_3$ solution serves to precipitate the aluminium completely and to dissolve the beryllium and uranium. This method of separation of aluminium and beryllium is a satisfactory one, when, as in the present case, only a small amount of aluminium is present; a large quantity of aluminium, however, retains almost completely a small quantity of beryllium. The $NaHCO_3$ solution must not be boiled for a long time, because, owing to the escape of CO_2 and the formation of free NaOH, the solution becomes alkaline enough to dissolve some aluminium. The reason for the great solubility of beryllium hydroxide in concentrated $NaHCO_3$ solutions is not known. H_2S is passed in to remove the iron, since a small amount of it remains dissolved in the concentrated $NaHCO_3$ solution; the filtrate is sometimes dark green even after repeated filtration, due to the presence of colloidal iron sulphide; to precipitate this, add a small amount of a ferrous salt (say 2 to 4 mgrms. Fe), shake, allow the mixture to stand several minutes, and then filter, finally passing more H_2S into the filtrate to make sure that the iron has been all removed.

2. The treatment with $K_4Fe(CN)_6$ described in *P. 58*d* gives a satisfactory separation of beryllium from uranium (and vanadium) provided the amount of beryllium in the solution does not exceed 20 milligrms. When more beryllium is present a gelatinous precipitate separates; on this account it is directed to use only a portion of the NH_4OH precipitate when it is large, but even in this case a small amount of uranium can still be detected, owing to the delicacy of the ferrocyanide test. Vanadium, if present owing to its having been precipitated as uranyl vanadate in the treatment with dilute $NaHCO_3$ (*P. 58*a*), remains with uranium and is precipitated as greenish yellow vanadyl ferrocyanide, but does not obscure the dark red colour of the uranium precipitate. It is not necessary to provide for the detection of vanadium at this point, since a large quantity always dissolves in the $NaHCO_3$ treatment.

(To be continued).

NOTICES OF BOOKS.

Matriculation Directory. June, 1908. London: University Tutorial Press, Limited. 1908.

This Directory to the Matriculation Examination of the University of London will be particularly useful to those students who are endeavouring to prepare themselves for the examination without the assistance of a tutor. It contains the papers set in the June examination with full solutions and criticisms of many of them, and the introduction makes valuable suggestions as to the most suitable text-books for the students' use. The advice given is, on the whole, sound, and the books recommended are undoubtedly reliable for the special purpose in view, although in many cases their value as general educational instruments may be inferior to that of others which have not been written so exclusively to meet the requirements of one particular syllabus.

The Theory of Ions. By William Tibbles, M.D., &c. London: Rebman, Limited. 1908.

This little book is a rather skilfully worked up summary of modern theories relating to the applications of physica chemistry to physiological problems, which of recent years have been made the subject of thorough investigation by many observers. The author's treatment of these problems contains nothing essentially new, nor is it even clear upon what definite facts he bases his opinion that life is a gradual development of an evolutionary character from dead to living matter, and that ions are the intermediate substance between living and non-living matter. However, the theories of physical chemistry are clearly stated in simple language, and the book will be interesting reading for those who take pleasure in studies of the origin of life and kindred problems.

CORRESPONDENCE.

MUST WOMEN BE DENIED FELLOWSHIP OF THE CHEMICAL SOCIETY.

To the Editor of the Chemical News.

Sir,—It is disturbing to learn, from his letter in the Chemical News of July 31st, that Mr. Droop Richmond did not mean what he set his hand to in the Petition concerning the Fellowship of the Chemical Society, namely, that he was "of opinion that the time had come when the Fellowship of the Society should be rendered accessible to women." More than this, to sign, as he explains he did, only to ascertain the wishes of the Society as to the admission of women, would seem to most people to be an unwarrantable act of aimless curiosity on the part of one who did not want to have them admitted. Let us hope, therefore, that Mr. Richmond may be wrong in believing himself to be not the only Fellow who signed as he did.

If Mr. Droop Richmond will refer to my letter of July 17th, he will see in it that I took no side on the question of admitting women to the Fellowship, and that, therefore, I could not, as he asserts, have given away one side or the other. He will further see that the whole of my letter was devoted to the endeavour (successful, I think) to show that, inadvertently, the procedure of the last Meeting of Council of the Society, acted upon in the way it was in the letter of the Honorary Secretaries, was calculated to affect unfavourably the success of the object of the petition. Mr. Richmond appears to have mistaken my position. For, I hold, not only that it is fair and proper, but that, in the interests of the Society and of chemistry, it is the duty of every Fellow to vote and to do all that he legitimately can to persuade others to vote his way on this matter of the admission of women. Acting upon this conviction, I wish now, sir, to be allowed to meet (what it was not my purpose to do in my first letter) the objections raised against granting the Fellowship to women.

The arguments for opening the Society to women are well stated in the Petition and in the Honorary Secretaries' letter, and such attempts as have been made to meet them do not seem to be serious enough to call for reply here. The real opposition to the admission of women has taken the form of arguments considered to outweigh in importance all that can be said in favour of admitting them. What appears to be the principal of these is put in the following paragraph.

The Fellowship of the Chemical Society would be an aid to a woman in getting employment as a chemist. Consequently, the possibility of gaining entrance to the Society on equal terms with men would encourage women to choose chemistry as the means of a living. But women ought not to be thus encouraged, because, not only is the work of the chemist of too arduous a nature for them, but they might on equal terms successfully compete for remunerative employment with men, many of whom support, or would support, a wife and children out of their earnings as chemists.

To me this argument is quite beside the mark, and, if it

were not, would still be indefensible, as I proceed to explain. It is beside the mark, since the object of the Society is the general advancement of chemical science. Such it is said to be expressly in the Charter, and such is what it has always been in the practice of the Society. Many times in past years, to learn that a candidate sought the right to append the letters F.C.S. to his name for "professional pursuits" or purposes led the voting Fellows to blackball him. It was, too, because the Society could not take up the professional interests of chemists that the Institute of Chemistry was founded. That Institute, by the way, whilst doing inestimably good service in looking after these interests, has not found it to be part of its duty to inquire into the sex of the candidate for its diploma.

The Chemical Society has no power under its Charter to issue in the form of its Fellowship, or in any other way, an authorisation to engage in chemical work, or a declaration of the competence of any one to engage in it. It has, therefore, had no consideration at all when granting its Fellowship, and can have none, for the question whether a candidate will be too feeble for "the arduous nature of chemical work." It has no consideration, either, for the question whether additional Fellows "may . . . find employment in view of the already overcrowded state of the profession," or whether its Fellowship must be restricted to those who have already, or are looking to have, a wife and children. It is utter silliness and rank trades-unionism to suggest that it has. The interests of the Society lie wholly in the advancement of chemical knowledge, and the welfare of the Society is measured by its success in this work, as shown by the ratio of increase in the number of its Fellows : *Multi pertransibunt et augebitur Scientia.*

This being so beyond disputes, it is to me an insolvable problem how a Fellow can believe it to be honourable and loyal to the Obligation he has signed to try to prevent a woman being a means, by becoming a Fellow, of "promoting the interests and welfare of the Society," in order thereby to impede her in a possible competition with himself, or some other man chemist, for a living. A Fellow has to vote as such and in no other capacity, and to be true to the interests of the Society, whatever he may deem to be his private interests.

Having, however, to face the fact that there are Fellows who are ready to put personal advancement before public responsibilities, I would point out to them, since they do recognise the claims of women to admission and express sorrow (some of them) at having to try to keep them out, that at the worst for themselves, it must be very many years before the competition of women for employment as chemists will be more than that of one woman against hundreds of men ; that it is the F.I.C. and the D.Sc., and not the F.C.S., which help chemists, women as well as men, to get appointments. The success already of women, even without the F.C.S., may be used against my argument that women chemists will for long be too few to be feared. But that is a dangerous objection to raise. I have special opportunity for judging and knowing about some University appointments filled by women chemists, and I am convinced that the interests of the Universities concerned would not be better served by replacing these chemists by men. Then, if I am right, what is the use of striving to keep women out of employment as chemists by handicapping them in their competition with F.C.S.'s ?

An argument advanced by Mr. Richmond against admitting women to the Fellowship is that they will be favoured on account of their sex by those estimating their fitness for it and the value of their papers. Supposing this to be true, the argument would only hold good were the F.C.S. a certificate of merit and ability, and it falls far short of being that. Moreover, the great majority of the present Fellows have no papers of theirs in the *Transactions* of the Society. Whilst, however, I do not see that the argument justifies Mr. Richmond in voting and asking others to vote against the admission of women into the Society, I admit that there is something in his objection to a woman chemist getting an unfair advantage in competition by means of exaggerated representations as to her chemical ability because of her sex. We may, however, count upon this abuse, if it exists, working out its own cure, and in the meantime we may war against it where we meet with it. Women themselves now cry out for no favour for sex, which is wise of them, and I am sure they mean it.

A more certain grievance for some men chemists against women chemists is that owing (not to the over-estimation, but) to the under-estimation of the value of women's abilities women may, and already do, have to render equivalent services for less pay than men chemists receive. Helplessly they may thus become unfair competitors with men. But this, of course, is a matter quite independent of that of the Fellowship.

The argument that the quantity of women's chemical work is insignificant when compared with that of the men is another having no application to the question of admitting women into the Society. Let there be but one woman waiting for admission whose chemical ability is beyond dispute, or even one who in the opinion of half-a-dozen Fellows signing her certificate, and that of the necessary majority at a meeting voting for her, why should she be forbidden the Fellowship because of there being, by supposition, not much in favour of other women ?

Yet another argument against women getting the Fellowship is that it would then be possible for a woman to become a Member of Council or other Honorary officer (there is, of course, already that admirably efficient, paid officer, Mrs. Dougal). Again, it has to be said, this is no just or allowable ground for excluding women. It is, besides, highly improbable that a woman Fellow would get office unless eminently qualified for it, but who then could object ? Her sex certainly would be no qualification for office. It is unreasonable to suggest that women, as a sex, will ever prove themselves to be better chemists than men, but unless they do there can be no chance of predominance of women in the Chemical Society.

I have, lastly, to refer to the difficulty in the way of letting women into the Society caused by the wording of the Charter, which is ambiguous. In the By-law on the election of a Fellow the candidate is spoken of as "he." Does this pronoun really signify here the sex of the candidate ? It is a familiar fact that the masculine pronoun is not only used for a male but for any member of the species, man, when the notion and distinction of sex are not involved. There were no women chemists when the Charter was granted, and no one will now contend that the pronoun was meant to be exclusive of women—that one condition of Fellowship was that the candidate must be male and not female. Provided that the Fellows as a body should not object, there would be no difficulty on this point, and "he" would be taken in its personal non-sexual sense. At worst, however, a Supplementary Charter, introducing only the additional words "or she," may be necessary, and an amended Charter is wanted on other grounds.

In conclusion, I venture to assert, as proved, that there is not one well-founded argument against the admission of women to the Fellowship of the Chemical Society, whatever may be the value of the trades-unionist arguments for persuading women not to attempt the practice of chemistry as a living, arguments, be it added, which apply equally to any more men who think of doing so.—Yours, &c.,

EDWARD DIVERS.

London, August 10, 1908.

CITRIC ACID.

To the Editor of the Chemical News.

SIR,—I should be glad if you could favour me with the whole or part of the following information on the production of citric acid desired by the Italian Government :—

1. List of firms in England using citrate of lime to produce citric acid, citrate of iron, and other products derived from citric acid.

2. Statistics of the annual importation into England of citrate of lime (Italian) from the various countries of origin for at least ten years.

3. The general contract conditions in buying citrate of lime (Italian) by English firms.

4. Half-yearly average prices of citric acid and of citrate of lime, and also average price for a period of ten years (Clause "C.I.F." and "F.O.B.," as the case may be).

5. Cost of production of 100 kgrms. of citric acid made with citrate of lime in England.

6. Purposes for which the products derived from citrate of lime in England are used, and quantity disposed of in England of such products.

7. List of importers of citrate of lime, concentrated juice, and raw juice.—I am, &c.,

L. Allatini,
Italian Consul General.

NITRIC ACID.

To the Editor of the Chemical News.

Sir,—For a small work I have in hand I wish to obtain the following particulars. Could you very kindly assist me to get them (or some) either by giving the figures or by saying where I could get them myself?

1. (*a*) Number of plants in Great Britain for making nitric acid. (*b*) Output —, total —, or for each if possible.

2. (*a*) Number of plants in Great Britain for making explosives which require nitric acid. (*b*) Tons of acid required if they were working normal full capacity.

3. (*a*) The different kinds of industrial uses for nitric acid. (*b*) Average amount they require (each kind of business) annually.

4. (*a*) Tons of nitric acid imported annually; (*b*) exported.—I am, &c.,

E. J. Deason.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Academie des Sciences. Vol. cxlvii., No. 2, July 13, 1908.

Action of Metallic Oxides (Irreducible) on Primary Alcohols.—Paul Sabatier and A. Mailhe.—Some metallic oxides which are not reducible act as catalytic decomposition agents upon primary alcohols at temperatures below 360°. Certain oxides, *e.g.*, manganous oxide, stannous oxide, decompose the alcohols, setting hydrogen free. Other oxides, *e.g.*, alumina and thoria, dehydrate alcohols; thus with ethyl alcohol they give ethylene, and some aldehyde condensation occurs; others give rise to both reactions simultaneously. The proportion of the two reactions varies according to the nature of the oxide and the temperature. The effect of raising the temperature is to increase the velocity of the two reactions, especially that of the aldehyde condensation.

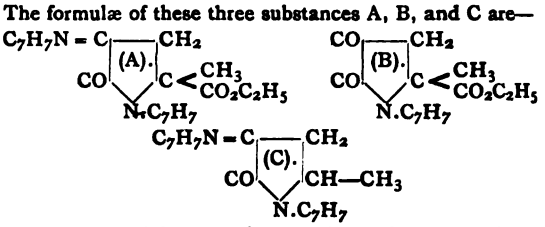
Mechanism of Synthesis of Nitrogen Cycles. Action of Ethyl Pyruvate on Paratoluidine.—L. J. Simon.—The action of ethyl pyruvate on paratoluidine may be represented by the equation—

$$CH_3{-}CO{-}CO_2C_2H_5 + 2C_7H_7{-}NH_2 = C_{22}H_{24}N_2O_3 + C_2H_5OH + 2H_2O.$$

$C_{22}H_{24}N_2O_3$ may be called A. Sulphuric acid causes the elimination of one molecule of paratoluidine — $C_{22}H_{24}N_2O_3 + H_2O = C_{15}H_{15}NO_4 + C_7H_7NH_2$. This new substance B is distinguished from A by its enol and ketone properties. With alcoholic potash A is saponified, yielding a third substance C.

$$C_{22}H_{24}N_2O_3 + 2KOH = C_{19}H_{20}N_2O + CO_3K_2 + C_2H_5OH.$$

The formulæ of these three substances A, B, and C are—



By the same original reaction another substance differing from A may be formed. The author has not yet definitely determined the constitution of this compound, which has the empirical formula $C_{24}H_{30}N_2O_4$, and which may be a polymerisation product of ethyl toluylpyruvate.

Sparteïn. Conversion of α-Methylsparteïn into Isosparteïn.—Amand Valeur.—The author has shown experimentally that by boiling a solution of *a*-methylsparteïn in dilute sulphuric acid until potassium permanganate is only very slightly reduced, and adding barium iodide, the sulphate of *a*-methylsparteïn isomerises, giving isosparteïn sulphomethylate, which can be isolated by slow evaporation *in vacuo*. The analogy between sparteïn and N-methylpiperidine is complete. N-methylpiperidine when subjected to Hofmann's reaction is transformed into an unsaturated base, N-dimethylpiperidine, the chlorhydrate of which forms the cyclic compound dimethylpyrrolidine chlormethylate; the ammonium hydrate corresponding to this last salt is decomposed, regenerating N-dimethylpiperidine. Similarly sparteïn by reactions of the same type yields α-methylsparteïn, isosparteïn, iodomethylate, and (by Hofmann's method) *a*-methylsparteïn.

Researches on Acid Sulphates of Potassium.—L. Arzalier.—The existence of the compound $SO_4K_2.\frac{1}{2}SO_4H_2$ cannot be proved by thermic methods. The amount of heat set free by the fixation of $\frac{1}{2}H_2SO_4$ to SO_4H_2 should be greater than $\frac{1}{2}$ of the amount of heat set free by the fixation of one molecule of sulphuric acid, whereas it is slightly smaller. This compound is formed only in the hydrated state in the presence of water, owing to the heat of hydration (+5·383). It seems *a priori* impossible that a more acid compound than $SO_4K_2.3SO_4H_2$ could exist, for the molecules of sulphuric acid which would add themselves on would have to set free less than 2·42 cals., which represents the heat of fixation of one molecule of sulphuric acid to the acid sulphate $SO_4K_2.SO_4H_2$. The substance thus obtained would be very unstable.

Analysis of Wines.—Paul Dutoit and Marcel Duboux.—By neutralising and measuring the electrical conductivity of the solution it is possible to determine simultaneously the sulphates, the total acidity, and the tanning matter in wine. A concentrated solution of alkali is added to the wine in small portions, and the conductivity is determined after each addition. The graphic representation of the values thus obtained gives a curve which is characteristic of each wine and each alkali. When baryta is used as alkali the curve assumes a definite form, and from the lengths of certain parts of the curve it is possible to determine accurately the constituents of the wine.

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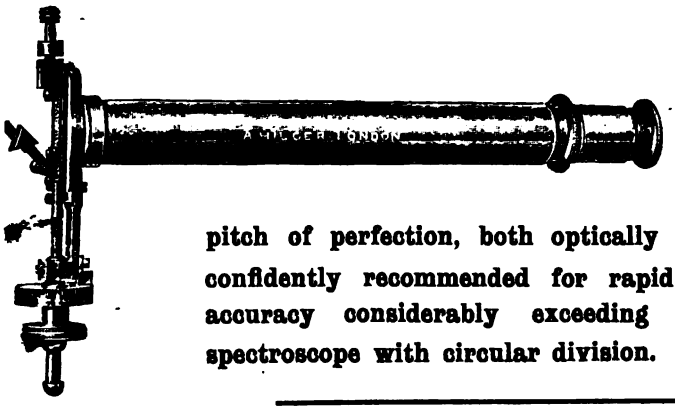

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ON THE ATOMIC WEIGHT OF CHLORINE.*

By EDWARD C. EDGAR, D.Sc., Assistant Lecturer in Chemistry in the University of Manchester.

SIX years ago Prof. Dixon and I began a research with the object of determining directly the weight of chlorine which combines with the unit weight of hydrogen. Our method was to burn a jet of hydrogen in an atmosphere of chlorine; hydrogen being stored and weighed in palladium, the chlorine being condensed and weighed as liquid. The number we obtained for the combining weight of chlorine was appreciably higher than that found indirectly by Stas, and still higher than that approved by the International Committee on Atomic Weights.

While this research was in progress, other determinations had been made bearing on the relative weights of silver, chlorine, and nitrogen, so that some modification in the accepted values of one or more of these elements appeared inevitable. The direct "joining up" of the two ends of the chain connecting hydrogen with chlorine thus became a matter of immediate importance. Since the method of burning one gas in an atmosphere of the other had been proved to be accurate within fairly narrow limits, I was encouraged to continue the investigation, and to modify the apparatus, with a view to eliminate some of the possible sources of error in the former series of experiments.

The most important source of error lies in the weighing of the hydrogen. To diminish this error the weight of hydrogen employed was doubled; and since Prof. Dixon and I found, when water was used to condense the hydrogen chloride formed in the flame, that some of the water vapour was decomposed by the free chlorine, I avoided this by burning a jet of chlorine in dry hydrogen, condensing the hydrogen chloride as it was formed in a tube dipped into liquid air. In some of the experiments the hydrogen chloride formed has been weighed. My experiments (concluded in 1907) agree closely with the results previously obtained in 1905. The method employed was briefly as follows:—

Hydrogen, made by the electrolysis of barium hydrate solution, and dried by potash and phosphorus pentoxide, was occluded, and weighed in palladium contained in a boro-silicate glass bulb. The chlorine, prepared by electrolysing fused silver chloride in a Jena glass vessel, was weighed as a liquid in a thick-walled boro-silicate glass bulb. Two ground joints attached these bulbs to a quartz combustion vessel, which was also connected with a vertical quartz tube, and with a steel bomb and a pump. After a thorough evacuation of the whole apparatus, the vertical limb of the combustion vessel was immersed in liquid air. Then the vacuous vessel was filled with hydrogen from the heated palladium bulb. Chlorine was ignited by a spark at the tip of a quartz jet, and continued to burn in the atmosphere of hydrogen with a fine needle-shaped flame. The endeavour was made so to regulate the gases as to maintain the flame until nearly all the chlorine weighed had been burnt. Then the supply of hydrogen was cut off. As the atmosphere became more attenuated the flame died away until, just before it went out, the chlorine was turned off. The hydrogen chloride, immediately after it was formed in the flame, was condensed as a snow-white solid by the liquid air surrounding the vertical limb of the combustion vessel; and a little chlorine, which had escaped burning, was also solidified. At the end of the combustion, the residual gas was extracted by the pump and analysed; it proved to be practically pure hydrogen.

Then the snow-white hydrogen chloride was allowed to evaporate. By passing the gas through a quartz tube filled with mercury vapour, the chlorine it contained was completely removed, and the purified hydrogen chloride passed on to a steel bomb immersed in liquid air, where it was condensed in six experiments, and successfully weighed in three; in the other three the bomb leaked. In two other experiments the gas was absorbed by water, and weighed as aqueous acid. The weights of hydrogen and chlorine burnt were obtained by subtracting from the total weight of each used the weight of hydrogen extracted by the pump, and the weight of chlorine caught by mercury vapour respectively.

The balance was specially made for this work by Oertling; even under the load of the steel bomb, weighing over 1000 grms., it gave very reliable readings. Each piece of apparatus weighed was tared with another of the same material, and of very nearly equal volume and weight, and the small weights used in the weighings were reduced to a vacuum standard.

	Hydrogen burnt, in grms.	Chlorine burnt, in grms.	Hydrogen chloride caught, in grms.	$\frac{\text{Chloride burnt}}{\text{Hydrogen burnt}}$	$\frac{\text{Hydrogen chloride caught} - \text{hydrogen burnt}}{\text{Hydrogen burnt}}$
1.	2·1452	75·5026	77·6469	35·196	35·196
2.	2·0387	71·7504	73·7880	35·194	35·194
3.	1·7762	62·5004	—	35·188	
4.	1·9935	70·1638	72·1565	35·196	35·196
5.	1·6469	57·9671	—	35·198	
6.	2·1016	73·9662	—	35·195	
7.	1·7254	60·7162	62·4401	35·190	35·189
8.	2·0885	73·4991	75·5859	35·192	35·191
		Mean		35·194 ± 0·0008	35·193 ± 0·0009

Above are set out the corrected weights of hydrogen and chlorine burnt in eight experiments, and the weights of hydrogen chloride caught in five.

If the atomic weight of hydrogen be taken as 1·00762, the mean values for the atomic weight of chlorine, calculated from the numbers in the accompanying table, are 35·462 ± 0·0008 and 35·461 ± 0·0009.

Dixon and Edgar, burning hydrogen in chlorine, found the equivalent of chlorine to be 35·463 ± 0·0019 from their nine experiments.

The concordance of the two sets of experiments is thus exceedingly close, and the number 35·462 may be taken as representing the result of the whole work.

On the other hand, Noyes and Weber (*Journ. Am. Chem. Soc.*, 1908, xxx., 13), by passing a known weight of hydrogen over weighed potassium chlorplatinate, noting the loss in weight of the salt, and condensing and weighing in water the hydrogen chloride formed, have recently obtained for the atomic weight of chlorine the mean number 35·452 ± 0·0008 (H = 1·00762).

In view of the promised re-calculation of the atomic

* Abstract of a Paper read before the Royal Society, June 25, 1908.

weights by the International Committee this year, I have not attempted to correlate my results with the recent determinations of silver, nitrogen, and chlorine.

THE ELECTROLYTIC PROPERTIES OF DILUTE SOLUTIONS OF SULPHURIC ACID.*

By W. C. D. WHETHAM, M.A. F.R.S., and H. H. PAINE, B.A., Trinity College, Cambridge.

PART I.—*By* W. C. D. WHETHAM.

1. *The Electrical Conductivities.*

THE first section of the present paper contains an account of a continuation of the work described in the *Proceedings of the Royal Society* (1905, A, lxxvi., 577), and a statement of the object of the investigation may be reproduced from that place :—

"If the measure of the electrical conductivity of a solution be divided by that of the concentration expressed in gramme-equivalents per unit volume, we obtain a quantity which may be called the equivalent conductivity of the solution. If the conductivity of the solvent used be subtracted from that of the solution, the corresponding quantity may be taken as giving the equivalent conductivity of the solute.

"As is well known, the equivalent conductivity of neutral salts, when dissolved in water, approaches a limiting value as the dilution is increased, and, in terms of the ionisation theory, this limiting value corresponds with complete ionisation.

"With solutions of acids and alkalis, however, the phenomena are different. As dilution proceeds, the equivalent conductivity reaches a maximum at a concentration of about a one-thousandth or a two-thousandth of a gramme-equivalent per litre, and then falls rapidly as the dilution is pushed farther.

"It has been supposed that this diminution of equivalent conductivity at extreme dilutions is due to interaction between the solute and the impurities which remain even in re-distilled water.

"Kohlrausch has given evidence to show that the chief impurity in water carefully re-distilled is carbonic acid," but the experiments described in the writer's former paper show that no appreciable change in the phenomenon is produced by boiling the water used as solvent repeatedly under low pressure, and re-admitting the air through potash bulbs. This process would remove a great part at least of the carbonic acid in the solvent, and it causes a large diminution in the conductivity of the water, but, on adding small quantities of sulphuric acid to solvent so purified, a conductivity curve of the normal type is obtained—the lowering of equivalent conductivity at great dilution is not affected appreciably. The first step in continuing the research consisted in repeating the process of boiling the solvent under diminished pressure, but in re-admitting air through bulbs containing dilute sulphuric acid as well as others containing potash. Any volatile alkaline impurity such as ammonia would thus partly be removed, and if such impurity were the cause of the phenomenon, the conductivity curves obtained by adding acid to the purified solvent would be modified. The results obtained are given in Table I.

As comparative numbers only were needed, the conductivities were expressed in arbitrary units—the reciprocals of the measured resistances in the cell employed. It will be seen that the usual phenomenon of a fall in the equivalent conductivity at great dilution is still evident, and the curve obtained by plotting $m\lambda$ and k_0/m is not appreciably different from that obtained when unboiled distilled water is used as solvent.

Thus the efforts made to remove residual ammonia are without result on the conductivity of solutions of sulphuric acid made up with the solvent so purified.

* A Paper read before the Royal Society, June 18, 1908.

Hence it seems that traces in the solvent neither of carbonic acid nor ammonia are competent to explain the peculiarities in the conductivity of dilute solutions of acids. As will be pointed out in Part II. of this paper, however, another possibility remains, namely, the presence in re-distilled water of traces of carbonate or bicarbonate of ammonia. Such an impurity might produce the effect observed both with acids and with alkalis, and the slight trace of it requisite might not be removed by treatment such as is described above. However this may be, it seems unlikely that the impurity which is usually assumed to be the cause of the phenomenon is competent to explain it.

2. *The Transport Ratios.*

If the phenomenon of the decrease in equivalent conductivity be not due to impurities in the solvent, it must be caused by some change in the conductivity of the solute itself. A decrease in conductivity must be accompanied by a decrease in the effective velocity of one or both of the ions. Since the phenomenon under investigation occurs with alkalis as well as acids, it seems likely that, if it be not due to impurities, it depends on the presence of the one ion in each of these classes of compounds which is identical with one of the ions of the solvent—in acids the hydrogen ion, and in alkalis the hydroxyl ion. Hence, on the supposition we are now making, it is probable that one ion would be affected in a different way to the other. We may expect that in solutions of acids, the hydrogen ion travels slower at great dilutions than at more moderate concentrations. Such a differential treatment of the ions would be made manifest by a determination of the migration constant or transport ratio of solutions of different concentrations. If an electric current be passed through a solution between non-dissolvable electrodes, solute is lost from the solution in the neighbourhood of both electrodes, and it is readily shown that the ratio of loss is equal to the ratio between the opposite ionic velocities. The ratio of the velocity of the anion to the sum of the velocities is taken as the transport ratio or migration constant. A decrease in the velocity of the hydrogen ion of acids, then, would cause an increase in the measured transport ratio.

The transport ratio usually has been measured by determining the concentration of the solution by chemical means before and after the passage of the current in the neighbourhood of the two electrodes. But, in the present case, the concentrations involved were far too small to be estimated chemically; it was necessary to devise a new method.

Now, in the measurement of electrical conductivity, we possess a means of determining the concentration of a dilute solution much more sensitive and accurate than any method of chemical analysis, and a method of experimenting was designed to utilise these advantages.

Fig. 1 shows a cell made of Jena glass. It consists of two vertical tubes connected horizontally through an inverted V, to the apex of which a quill tube of glass is fused. To this quill tube is fixed a piece of rubber tubing, and by a slight increase of air pressure the level of the liquid in the inverted V can be depressed so as to separate the liquid into two parts, one confined to each vertical limb of the apparatus.

In each vertical tube are placed two platinum electrodes, carried by glass tubes through the ends of which stout platinum wires are sealed. By measuring the resistance between these electrodes, the specific resistance of the solution round them can be determined, while, if each pair of electrodes is connected electrically together, a current may be passed through the whole apparatus from one pair of electrodes to the other when the liquid fills the inverted V tube as well as the vertical limbs.

The electrodes in the side tubes lie just above the junctions with the connecting tube. By this arrangement, the danger of convection currents, leading to a mixture of the anode and cathode portions of the solution, is avoided, for, in the cathode region, where the concentration diminishes, the lighter liquid rises towards the surface, while the denser anode liquid has no tendency to pass into

TABLE I.

No. of solution.	Concentration (grm.-equivalents per 1000 grms. of solution = m.	$m^{\frac{1}{2}}$.	Conductivity at 18° C. in arbitrary units = k.	Conductivity less that of solvent = k_0.	Equivalent conductivity of sulphuric acid = k_0/m.
0.	0·0	—	$1·041 \times 10^{-5}$	—	—
1.	$4·812 \times 10^{-4}$	0·03638	$1·800 \times 10^{-4}$	$1·696 \times 10^{-}$	3·526
2.	$9·158 \times 10^{-4}$	0·04506	$3·542 \times 10^{-4}$	$3·438 \times 10^{-4}$	3·757
3.	$1·983 \times 10^{-3}$	0·05831	$7·817 \times 10^{-4}$	$7·713 \times 10^{-4}$	3·890
4.	$3·579 \times 10^{-3}$	0·0710	$1·413 \times 10^{-3}$	$1·403 \times 10^{-3}$	3·936
5.	$6·523 \times 10^{-3}$	0·0935	$2·543 \times 10^{-3}$	$2·533 \times 10^{-3}$	3·910

the rising connecting tube, and sinks to the bottom of the side limb of the apparatus. The influence of diffusion was shown to be negligible in the course of the experiments, for identical results were obtained in similar experiments of long and short duration, in which the effects of diffusion would differ.

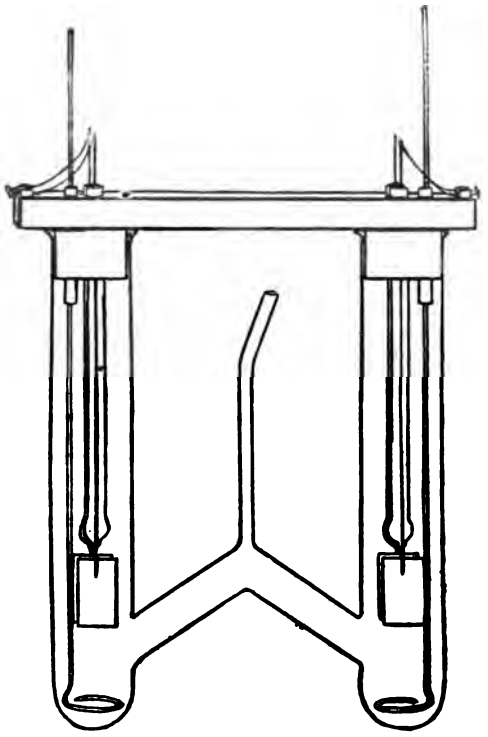


FIG. 1.

The mode of procedure is as follows :—A solution of pure potassium chloride is made up of a convenient strength by weighing out the salt. This solution is diluted by adding a known weight of water. The resistance of the solution so prepared, when placed in the cell, is measured between each pair of electrodes. From the specific resistance of potassium chloride solutions, as given by Kohlrausch, this gives the cell constant of each end of the apparatus, treating each vertical limb as a separate resistance cell. After careful washing, the apparatus is then filled with a solution of sulphuric acid, the concentration of which is known to be approximately that required. By measuring the resistance between each pair of electrodes, two values for the exact concentration may then be calculated from Kohlrausch's tables.

The apparatus is now ready for a migration experiment. A current from a battery of 50 or 100 small storage cells is passed through the apparatus from one pair of electrodes to the other, each pair in this part of the work being used as a single electrode. The strength of the current is determined by passing it through a standardised shunted galvanometer, and thus the total amount of chemical decomposition calculated from the known values of the electro-chemical equivalents. After a definite time has elapsed, the circuit is broken, the two parts of the solution are separated by increasing the pressure of the air in the inverted V tube, and measurements are taken of the resistance between each pair of electrodes as before. The change in concentration in the solution round each electrode may thus be estimated, and, by extracting and weighing each part of the solution, the total change in contents of acid calculated.

In a solution of sulphuric acid, the ions are hydrogen and the sulphion group SO_4. Hydrogen is liberated as such at the cathode, but the sulphion reacts with the water to form sulphuric acid and oxygen, the latter escaping. Thus when 1 grm.-equivalent of hydrogen is evolved, 1 grm.-equivalent of acid is re-formed at the anode by the secondary action of the sulphion and the water. But, besides this change, that due to migration must be considered. If the cation of any salt solution drifts one way with a velocity u, and the anion the other with a velocity v, the amount of separation in unit time is $u+v$. In accordance with the principle of migration, there is a loss of salt measured by v at the cathode, and a loss measured by u at the anode. But in our case there is also a gain of $u+v$, equal to the total amount of decomposition, at the anode. Thus the resultant gain at the anode is $u+v-u$ or v, and the resultant loss at the cathode is also v. Hence, by determining the loss and gain, we get two independent values for the same quantity, and the concordance between them gives a preliminary test of the success of the apparatus and method.

Finally, a knowledge of the value of the current and its time of flow gives $u+v$, and enables us to calculate $v/(u+v)$, the transport ratio.

After some changes, the apparatus in its final form gave satisfactory results as tested by the agreement between the loss of acid at the cathode and the gain at the anode. Details of one experiment may be given as an example.

Resistance before passage of current .. { Anode vessel—7102, 7106, 7112 = 7107 mean in ohms. Cathode vessel—6252, 6258, 6264 = 6258 mean in ohms.

Current from 50 cells (= 100 volts) passed for fifteen minutes—

Resistances.. { Anode vessel .. 8316, 8263, 8296 = 8297 Cathode vessel . 5452, 5461, 5464 = 5462

Weight of solutions in anode vessel .. = 53·25 grms.
Weight of solutions in cathode vessel . = 57·00 „

From these results it follows that the conductivity of the solution before the passage of the current was $1·408 \times 10^{-3}$.

After the passage of the current the conductivity of the anode solution was $1·614 \times 10^{-3}$, and of the cathode solution $1·219 \times 10^{-3}$.

The concentration of the original solution was $4·042 \times 10^{-3}$ grm.-equivalent per 1000 grms. From the known curve between concentration and equivalent conductivity, the concentration of the final anode and cathode solutions was estimated as $4·575 \times 10^{-3}$ and $3·546 \times 10^{-3}$ respectively. Then, from the observed total masses of their solutions, the total gain and loss in contents was calculated as

$2{\cdot}843 \times 10^{-7}$ grm.-equivalent and $2{\cdot}831 \times 10^{-7}$ grm.-equivalent respectively.

A rough estimate of the current gave $1{\cdot}491 \times 10^{-4}$ ampère, and, as the total number of grm.-equivalents liberated, $1{\cdot}390 \times 10^{-6}$. Thus the transport ratio is approximately $0{\cdot}2836/1{\cdot}390 = 0{\cdot}205$.

No stress can be laid on this last result, as the method of measuring the current was not finally adopted as satisfactory, but the concordance between the gain of acid at the anode and the loss at the cathode showed that, so far, the method was successful.

At this point the pressure of other duties prevented the writer from continuing the experiments, but, by the aid of the Government Grant Committee of the Royal Society, the services of Mr. H. H. Paine were secured. The work described in Part II. of this paper is Mr. Paine's; the present writer only exercised a general supervision over the course of the research.

(To be continued).

SOME EFFECTS OF SUNLIGHT UPON COLOURLESS GLASS.

By ROSS AIKEN GORTNER.

Within recent years a number of instances have been reported from various parts of the world concerning the violet coloration of glass when exposed to the sunlight (Crookes, Chemical News, 1905, xci., 73; Avery, *Journ. Am. Chem. Soc.*, 1905, xxvii., 909; Simpson, Chemical News, 1905, xci., 236; Rueger, *Journ. Am. Chem. Soc.*, 1905, xxvii., 1206; Alway and Gortner, *Am. Chem. Journ.*, 1907, xxxvii., 1).

Fischer (*Ber.*, 1905, xxxviii., 946) has induced the same change in colourless glass, containing a comparatively small proportion of manganese, by exposure to the rays of a mercury lamp, thus showing that the change is due to the ultraviolet rays.

Up to the present time only one instance, which was observed by the writer (*Am. Chem. Journ.*, 1907, xxxvii., 1), has been reported indicating the time of exposure actually required to produce the change in the colour of the glass.

On July 9, 1906, twenty-two specimens of glass were firmly fastened to a board by means of aluminium wire, and the board exposed upon the slate roof of a building at Lincoln in a position facing the south and inclined at an angle of about 45°. The spot selected was about 75 feet above the ground and was exposed to the direct rays of the sun for the entire day excepting, perhaps, in June and July for an hour before sunset, when a low tower on the west cast a shadow on the board. The specimens, when on the building, were at an elevation of about 1200 feet above sea level.

Specimens 1 to 12 inclusive were rectangular four-ounce bottles of the same make, and were obtained from a local dealer. They were exposed, filled with various substances, Nos. 1 to 6 being filled with chalk coloured, respectively, with picric acid, indigo, methyl violet, "wasserblau," malachite green, and sodium eosin. No. 7 was filled with manganese dioxide, No. 8 with lampblack, No. 9 with sodium eosin solution, No. 10 with potassium permanganate crystals, No. 11 with chalk, while No. 12 was exposed empty. Nos. 7, 8, and 10 were filled with their respective contents in order to determine what truth there might be in the theory of Chas. Rueger that glass might become "violet by lying for some time in the proximity of bodies heavily charged with manganese" (*Journ. Am. Chem. Soc.*, 1905, xxvii., 1206). No. 13 was a bottle of a different make and was exposed filled with potassium chromate crystals. No. 14 was the base of a broken goblet which was found upon the street, and which as yet showed no trace of violet coloration, although it might have been exposed to the sunlight for some time. No. 15 was a small glass funnel, No. 16 a small round solution bottle, No. 17 a 50 cc. Erlenmeyer flask of Jena glass, No. 18 an ordinary round 50 cc. flask, No. 19 a small homœopathic vial of American manufacture, No. 20 a piece of hard glass tubing, and Nos. 21 and 22 pieces of soft glass tubing. Nos. 15, 16, 17, 20, 21, and 22 were secured from the "Vereinigte Fabriken," Berlin.

One month after exposure the specimens were examined and it was found that Nos. 13, 14, 21, and 22 had become slightly violet, No. 13 being of so decided a violet tinge that the coloration was noticeable several feet away.

The specimens of glass then remained undisturbed until the 21st of March, 1907, when they were again examined. At that time they all showed some violet coloration excepting Nos. 15, 16, 17, and 20, which still remained colourless. Nos. 13 and 21 had by this time become intensely violet. No. 22 could not be found, evidently having been blown away by the wind.

On May 31, 1907, the board was removed from the roof, and the bottles, after being cleaned, were given a close inspection, with the following result:—No. 1 was distinctly violet on the upper face, less violet on the sides, but still enough so that the coloration was plainly visible, and almost colourless on the back. Nos. 2, 4, 5, 6, and 11 differed in no way from No. 1. No. 3, which had contained chalk coloured with methyl violet, was of a deeper colour on the upper face and sides and was also of a faint violet on the back. No. 7 (filled with manganese dioxide) and No. 8 (filled with lampblack) were indistinguishable from each other, being less violet on the surface and sides than the preceding numbers and showing almost no colour on the back. No. 9 had been broken by the freezing of its contents; the violet tinge on the face and sides was pronounced, and on the back less so. No. 10, containing potassium permanganate crystals, was the least coloured of the series 1—12, being slightly less violet than Nos. 7 and 8. There was a faint violet tinge upon the upper face and almost no colour whatever upon the sides and back. No. 12, being empty, was of a uniform violet throughout, and the colour could not be distinguished from that of the face of No. 1. No. 13 was a very intense violet upon the face and sides and of a deep violet on the back. This is the best specimen of violet-coloured glass that I have been able to secure in Nebraska, although it is surpassed in intensity of coloration by some specimens from the steppes of Canada and from New Mexico. (The specimens from New Mexico were secured from Dr. Avery). No. 14 was of a deep violet throughout, being more intense in colour than No. 12 and much less intense than No. 13. The shade was slightly deeper than on the back of No. 13. Nos. 15, 16, 17, and 20 showed no trace of violet coloration. Nos. 15, 16, and 20, as will be shown later in the table of analyses, contained no manganese, and No. 17, although containing manganese, was not coloured violet by the action of the sunlight. An explanation for this may be found in the composition of Jena glass which may prevent the necessary chemical change from taking place. Fischer met with similar results when Jena glass was exposed to the rays from a mercury lamp (*Ber.*, 1905, xxxviii., 946). Nos. 18 and 19 were distinctly violet throughout. No. 21 was distinctly violet when seen from the side, even at a distance of several feet, and when viewed lengthwise the violet coloration was very intense, being but a shade lighter than No. 13.

In order to obtain the manganese content, the method of H. Noll was employed (*Centralbl.*, April 17, 1907, i., 1223). Portions of the specimens were ground to a powder, fused with sodium and potassium carbonates, dissolved in dilute hydrochloric acid, precipitated with ammonia and bromine water, and allowed to stand for twelve hours. The precipitate was then filtered out, washed, and treated with 1 grm. potassium iodide and 4 cc. concentrated hydrochloric acid. After standing for five minutes the iodine set free was titrated with N/100 sodium thiosulphate. From the iodine set free the manganese is calculated.

No. of sample.	Description of sample.	Per cent of manganese.	Colour after one month.	Colour after eight months.	Colour after eleven months.
15.	Small funnel (Verein. Fabrik.)	0·000	None.	None.	None.
16.	100 cc. bottle (Verein. Fabrik)	0·000	None.	None.	None.
20.	Hard glass tubing (Verein. Fabrik.)	0·000	None.	None.	None.
14.	Base of goblet	0·049	Slight.	Distinct.	Deep.
17.	50 cc. Erlenmeyer flask (Jena glass)	0·072	None.	None.	None.
1.	Bottle with chalk coloured with picric acid	0·075 to 0·087	None.	Light.	Distinct.
2.	Do. do. indigo			Light.	Distinct.
3.	Do. do. methyl violet			Distinct.	Deeper than No. 1.
4.	Do. do. "wasserblau"			Light.	Distinct.
5.	Do. do. malachite green			Light.	Distinct.
6.	Do. do. sodium eosin			Light.	Distinct.
7.	Bottle with manganese dioxide			Pale.	Light.
8.	Bottle with lamp-black			Pale.	Light
9.	Bottle with sodium eosin solution			Distinct (broken).	Distinct.
10.	Bottle with potassium permanganate crystals			Faint.	Very light.
11.	Bottle with chalk			Distinct.	Distinct.
12.	Bottle, empty			Distinct.	Distinct.
19.	Homœopathic vial (American manufacture)	0·14	None.	Distinct.	Deep.
18.	Round flask (American 50 cc.)	0·17	None.	Distinct.	Deep.
21.	Soft glass tubing	0·20	Pale.	Distinct.	Deep.
13.	Bottle with potassium chromate crystals	0·34	Distinct.	Deep.	Very deep.
22.	Soft glass tubing	(?)	Distinct.	Lost.	Lost.

The colour of the beads which were obtained by the fusion ranged from a deep purple in No. 13 to colourless in Nos. 15, 16, and 20. The range of coloration was in the following order:—13, 11, 19, 18, 21, 14, and 17, 13 being the most coloured and 17 the least. The beads obtained from Nos. 1 to 12 were indistinguishable, and therefore No. 11 was selected to represent the series.

The accompanying table gives the percentage of manganese and shows the relation of this to the change in colour on the face of the specimen. "Slight" means slightly violet, "pale" means pale violet, &c.

The influence of the exposure of glass is well illustrated by the colour of the basement windows of a church near Lincoln which has been built for five years. The windows are of pressed glass, and on the south and west sides are of such a distinctly violet colour as to be easily noticeable a hundred yards away, while on the north and east sides there is almost no coloration. When closely examined, the violet coloration is so intense as to be comparable only to the deeper coloured glass found on the high plains of Canada. The coloration is as deep as that of No. 13 although the manganese content is only 0·126 per cent, the deep colour being probably due to the length of exposure.

Beside the specimens of violet-coloured glass secured in this vicinity, the writer has found many scattered over eastern and northern Nebraska, and has received some from the second steppe of Canada. The latter came from southern Saskatchewan at an elevation of about 2000 feet.

Summary.

Glass containing a relatively large percentage of manganese may show a violet coloration when exposed to sunlight for a period of less than a month, the degree of coloration being proportional to the manganese content.

Exposure of less than a year will produce a violet coloration in most glasses containing only a small percentage of manganese, but this coloration will become more pronounced when the time of exposure is increased.

Should a glass be coloured violet by the action of sunlight, proof is furnished that the glass contains manganese. However, not all glasses containing manganese are coloured violet by exposure to sunlight, as is shown by the Jena Erlenmeyer.

A background or subsoil containing manganese or ores of manganese will have no influence upon the coloration of glass lying upon it.

A violet background is more favourable to the action of the ultraviolet rays than other colours: white, yellow, blue, and red act alike and seem to have no effect, while brown and black seem to have a retarding effect.—*American Chemical Journal*, 1908, xxxix., p. 157.

THE ESTIMATION OF CHROMIUM AS SILVER CHROMATE.

By F. A. GOOCH and L. H. WEED.

IT has been shown by Autenrieth (*Ber.*, xxxv., 2057) that when chromic acid is added to a boiling solution of silver nitrate, or when a soluble chromate or dichromate is added to a solution of silver nitrate previously acidified with nitric acid, or when silver chromate is treated with nitric acid, silver dichromate is formed; and that, on the other hand, it is silver chromate which is precipitated when silver nitrate in excess is added to a solution of a soluble dichromate, cold or hot, the reaction proceeding according to the equation—

$$4AgNO_3 + K_2Cr_2O_7 + H_2O = 2Ag_2CrO_4 + 2KNO_3 + 2HNO_3.$$

The characteristics of both silver dichromate and silver chromate have recently been summarised and further studied by Margosches (*Zeit. Anorg. Chem.*, xli., 68; l., 231), but so far as we know there is in the literature no account of procedure for the exact quantitative determination of either chromium or silver based upon the characteristics of either of these salts. The solubility of silver dichromate in water and in ordinary solutions is such as to preclude the use of this substance as the final product of a quantitative process depending upon precipitation. The solubility of silver chromate in a moderately large volume of water is not inconsiderable, and the solvent action of free acid, even acetic acid in quantity, is marked. We have found, however, that the precipitation of silver chromate is practically complete in a solution only faintly acid with acetic acid and in presence of a large excess of silver nitrate. If such a precipitate is collected in the filtering crucible and washed with a dilute solution of silver nitrate, until no other impurities remain, silver chromate does not dissolve, and the excess of silver nitrate may be removed by the cautious use of water without appreciable effect upon the precipitate. The present paper has to do with the determination of chromium as silver chromate.

In the experiments of which the details are given in the table, the general treatment just described was put to the practical test. Given amounts of potassium dichromate were weighed out and dissolved in hot water, as in

Experiments 11 and 12, or given amounts of a solution of potassium dichromate of known strength were run from a burette into a beaker and heated to boiling, as in Experiments 1 to 10. To the hot solution of the dichromate was added, drop by drop, a solution of silver nitrate in considerable excess, and the mixture was again brought to the boiling-point. Ammonium hydroxide was added until the clear liquid became colourless and turned litmus-paper blue; then acetic acid was added cautiously until the reaction of the solution was distinctly acidic to litmus. After standing for at least half-an-hour the precipitate was filtered off on asbestos in a perforated crucible, washed first with a dilute solution of silver nitrate and then with 20 cc. to 30 cc. of distilled water applied in small portions, and dried with gentle heating to a constant weight. In Experiments 1 to 10 the drying was done in an air-bath heated to 135°; in Experiments 11 and 12 the crucible and precipitate were heated gently over the free flame. In Experiments 7 and 8 the precipitation was made in presence of 5 grms. of ammonium nitrate, and in 9 and 10 in presence of 5 grms. of sodium nitrate, to test the effect of each of these substances upon the process. In no case did the filtrate, with the washings, show by the lead acetate test the presence of a chromate.

The Precipitation of Silver Chromate.

	$K_2Cr_2O_7$ taken.	$AgNO_3$ used in precipitation.	Volume at precipitation.	Ag_2CrO_4. Found.	Theory.	Error.
	Grm.	Grm.	Cc.			
1.	0·0921	0·4248	100	0·2072	0·2076	−0·0004
2.	0·0921	0·4248	100	0·2073	0·2076	−0·0003
3.	0·0921	0·4248	100	0·2075	0·2076	−0·0001
4.	0·0921	0·4248	100	0·2074	0·2076	−0·0002
5.	0·0921	0·4248	100	0·2075	0·2076	−0·0001
6.	0·0921	0·4248	100	0·2073	0·2076	−0·0003
7.	0·0921	0·4248	100	0·2073	0·2076	−0·0003*
8.	0·0921	0·4248	100	0·2075	0·2076	−0·0001*
9.	0·0921	0·4248	100	0·2080	0·2076	+0·0004†
10.	0·0921	0·4248	100	0·2070	0·2076	−0·0006†
11.	0·5801	3·0000	150	1·3087	1·3082	+0·0005
12.	0·7352	3·0000	200	1·6573	1·6574	−0·0001‡

* The precipitation was made in presence of 5 grms. of NH_4NO_3.
† The precipitation was made in presence of 5 grms. of $NaNO_3$.
‡ An excess of 1 cc. of 40 per cent acetic acid was added before filtering.

From the results of these experiments it is apparent that accurate determinations of chromium taken as the chromate or dichromate may be secured by precipitating silver chromate in presence of an excess of silver nitrate, making the solution ammoniacal and then faintly acid with acetic acid, transferring the precipitate to the filtering crucible, washing with a dilute solution of silver nitrate, and, after other soluble impurities have been removed, finishing the washing with small amounts of water applied in successive portions.—*American Journal of Science*, xxvi., p. 85.

NOTES ON A NEW PRIMARY VOLTAIC CELL OF THE DANIELL TYPE.

By JAMES STRACHAN.

THE voltaic cell about to be described was devised by the writer about eight years ago, and was set up for the first time in the chemical laboratory of George Heriot's Hospital. While experimenting with various chlorinated compounds, such as oxychlorides, perchlorides, and tetrachlorides, with regard to their suitability as depolarisers in primary voltaic cells, tetrachloride of lead ($PbCl_4$) was found to be a powerful and efficient depolariser. Since that time I have proved on various occasions the usefulness of this cell in the laboratory, and apart from its practical value, it is also of great theoretical interest as an illustration of electro-chemical depolarisation.

Name of cell.	Anode.	Excitant.	Cathode.	Depolariser.	E.M.F. (volts).	Advantages.	Disadvantages.
De la Rue	Zinc	NaCl or $ZnCl_2$	Silver	AgCl	1·3—1·4	Compactness.	Expense.
Von Helmholtz . .	Zinc	$ZnCl_2$	Mercury	Hg_2Cl_2	1·0	Constant action.	Expense.
Becquerel (modified)	Zinc	HCl	Lead	$PbCl_2$	0·5	Cheapness.	Easily polarised.
Papet	Iron or tin	Fe_2Cl_6 or $SnCl_4$	Carbon	Fe_2Cl_6 or $SnCl_4$	0·4—0·7	Simplicity and constant action.	Low E.M.F.
Maron	Zinc	H_2SO_4	Carbon	Fe_2Cl_6	1·5	Recuperates when exhausted.	High resistance.
Gerardin	Iron	Water	Carbon	Fe_2Cl_6+HCl+HNO_3	1·0	Cheapness.	High resistance chlorine evolved.
Niaudet	Zinc	NaCl	Carbon	CaCl(OCl)	1·5	Cheapness.	High resistance, and evolves fumes of chlorine.
Fitch	Zinc	NH_4Cl	Carbon	$NaClO_3$+$KClO_3$	1·1	Simplicity.	Easily polarised.
Heriot, No. 1. . .	Zinc	NH_4Cl+$ZnCl_2$+HCl	Carbon	$PbCl_4$ in HCl	1·5	Constant action and low resistance.	Odour of chlorine.
Heriot, No. 2. . .	Iron	NH_4Cl+$ZnCl_2$+Fe_2Cl_6	Carbon	$PbCl_4$ in HCl	0·95	Cheapness.	Odour of chlorine.

The new cell is of the Daniell type, and in it the copper element is replaced by carbon, the copper sulphate by tetrachloride of lead, and the dilute sulphuric acid, surrounding the amalgamated zinc, by dilute hydrochloric acid, or other excitant.

The action of nascent hydrogen upon the tetrachloride of lead is similar to that which takes place upon the copper sulphate in a Daniell cell, metallic lead being deposited in place of copper, and hydrochloric acid formed in place of sulphuric acid, as in the following equations:—

Daniell cell: $H_2+CuSO_4=Cu+H_2SO_4$.
Heriot cell: $2H_2+PbCl_4=Pb+4HCl$.

The cell may be conveniently arranged as in the accompanying sketch. Within an outer stoneware jar, O, are placed a number of carbon rods, C C, which are connected electrically by the heavy leaden cover, L, to form the negative element. The rods may consist of stout arc-light carbons, the ends of which are coppered by electro-deposition previous to casting the leaden cover around same. In the centre of the cover is a circular aperture which admits neatly the insertion of the porous earthenware pot, P, within which stands the positive element, an amalgamated zinc cylinder, Z. An acid solution of lead tetrachloride surrounds the carbon rods, and the zinc is immersed in a suitable excitant, which may consist of dilute hydrochloric acid (1 in 7 of water), zinc chloride (5 to 10 per cent solution), or ammonium chloride (15 to 20 per cent solution). Mixtures of these excitants may be employed, and the following gives good results:—

Ammonium chloride	100 grms.
Zinc chloride	10 „
Hydrochloric acid	20 cc.
Water to 1 litre.	

Unfortunately, lead tetrachloride is not an article of commerce, and the preparation of the depolariser presented at first some difficulties. The usual methods of preparing $PbCl_4$ solution in the laboratory are:—

1. Passing chlorine gas into hydrochloric acid containing freshly precipitated lead dichloride in suspension: $PbCl_2+Cl_2=PbCl_4$.
2. Dissolving lead peroxide in cold concentrated hydrochloric acid: $PbO_2+4HCl=PbCl_4+2H_2O$.

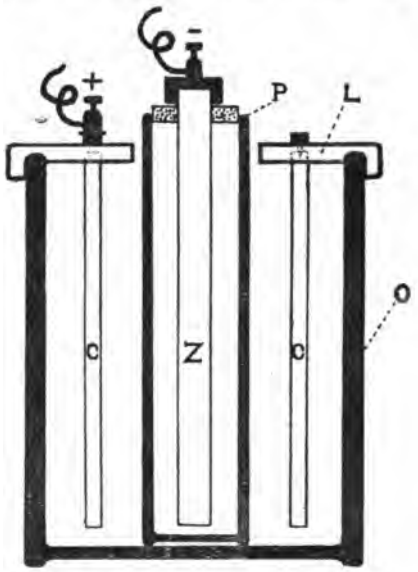
The latter method is the simpler, but it is both dangerous and disagreeable to perform, as the action is incomplete, $PbCl_2$ being precipitated with the evolution of much chlorine. After some experimenting it was found that freshly precipitated lead peroxide dissolves quite readily in cold strong hydrochloric acid. The precipitate of PbO_2 may be best prepared by dissolving 100 grms. of lead acetate in 500 cc. of cold water, and then adding a strong solution of calcium hypochlorite, prepared by lixiviating 110 grms. of fresh bleaching-powder in 1 litre of water, and decanting. Yellow PbO is at first precipitated, but after a few hours it becomes completely oxidised to the puce-brown peroxide. The heavy PbO_2 sinks to the bottom, and the bulk of the supernatant liquor may be poured away. The remaining pasty mass of peroxide is then stirred up to a creamy consistency and added gradually to about 500 cc. of cold concentrated hydrochloric acid in a Winchester quart bottle. The latter is kept cold by immersion in running water, and is gently shaken after each addition of the peroxide. The PbO_2 dissolves readily with evolution of a small amount of chlorine, forming an acid liquor of a bright orange-yellow colour, which is the depolariser as used in the cell.

A much simpler method of preparing the solution of tetrachloride suggested itself, and was found to work well in practice. 100 grms. of lead acetate were dissolved in 300 cc. of cold water, and 110 grms. of fresh bleaching-powder mixed with same in a strong pressure bottle. The mixture rapidly became brown in colour, due to the formation of lead peroxide. When quite cold about 700 cc. of strong commercial hydrochloric acid were added, and the pressure bottle rapidly sealed. During the ensuing action the bottle was cooled in running water. The brown mixture dissolved rapidly to form a solution of calcium chloride and lead tetrachloride, which was used as the depolarising fluid. This operation is rather a dangerous one to perform, and care must be taken to guard against the sudden evolution of the poisonous chlorine. When a glass pressure bottle is employed, the mixture of lead acetate and bleaching-powder should be made in a mortar with enough water to form a creamy paste, and the latter added gradually to the hydrochloric acid contained in the bottle, sealing and agitating after each addition. Large quantities of lead tetrachloride could be made very easily by this method by dissolving the lead acetate and chlorinated lime in lead-lined cylinders of wrought iron.

The acid solution of lead tetrachloride should not be exposed to sunlight, as it is then decomposed, oxygen being evolved and crystalline plates of $PbCl_2$ precipitated:—

$$PbCl_4+H_2O=PbCl_2+2HCl+O.$$

The solution is also decomposed very readily by many organic substances, since it possesses the oxidising properties of chlorine water in an intensified degree. In the presence of oxidisable organic matter, $PbCl_4$ yields



one-half of its chlorine, and crystalline needles or rhombs of $PbCl_2$ are precipitated. As a depolariser it is rapid, efficient, and constant in its action. On a closed circuit this cell cannot be polarised while any of the $PbCl_4$ remains in the solution around the negative element. In these respects it resembles the Daniell cell, but possesses a higher electro-motive force and a much lower resistance. Lead tetrachloride has a rather serious disadvantage, however, in the powerful odour of chlorine that attends its preparation and use. The cell must be charged either in the open air or in a draught chamber. While in use, the escape of chlorine fumes is entirely prevented by the closely-fitting leaden cover of the cell, the space between the porous pot and the inner edge of the cover being sealed, for the time being, with soft paraffin-wax. After the cell has been in action for some time the carbon rods become coated with a spongy deposit of metallic lead, which is easily removed by scraping with a spatula. Through continued action the pores of the carbon rods sometimes become filled up with coherent metallic lead. This may be removed by dipping the carbons into strong nitric acid. When the cell is required to give as large an output of current as possible, the excitant should be strengthened by the addition of hydrochloric acid (1 part to 4 parts of water), the zinc

should be heavy and well amalgamated, and the carbon rods should be replaced by several carbon plates surrounded by tightly-packed granules of the same material. With the exception of silver chloride, lead tetrachloride is the most efficient depolariser, using chlorine in union with a metallic base.

The accompanying table shows in comparison various cells, using such depolarisers and their relative values.

As shown in the table, iron may be employed in the new cell in place of zinc, when the E.M.F. drops from 1·5 to 0·95. When iron is used as the positive element, the hydrochloric acid should be left out of the excitant, and the latter be mixed as follows:—

Ammonium chloride	100 grms.
Zinc chloride	10 „
Ferric chloride	10 „

Water to 1 litre.

There is much more local action with iron than with zinc, and this action appears to be much less when tinned iron is employed.

I am much indebted to Mr. James D. Fulton, M.A., B.Sc., of Edinburgh, for the following results of experiments determining the E.M.F. of this cell, using zinc and iron as the positive elements with various excitants; and carbon as the negative element immersed in the acid solution of $PbCl_4$:—

Excitant used.	Electromotive force (volts).	
	With zinc.	With iron.
Hydrochloric acid, dilute	1·41	0·83
Zinc chloride solution	1·22	0·73
Ammonium chloride solution ..	1·51	0·95

The above figures were obtained by comparison with a standard Daniell cell, E.M.F. 1·111, and were further checked with a single cell of an accumulator in place of the standard, consistent results being obtained.

Chemical Laboratory, Ballyclare, Co. Antrim.

THE EXISTENCE OF PLATINUM METALS IN CHEMICALS USED FOR ASSAYING.*

By JOHN G. ROSE, Government Analyst, Cape Town.

DURING the past twelve months upwards of fifty samples of rock have been assayed in the Government Analytical Laboratory, Cape Town, for platinum, in all but two cases with negative results. These two exceptions having yielded such minute traces it was a matter for considerable surprise when a sample of acid dolerite from Stutterheim in the Eastern Province, and a sample of white quartz from Swellendam, in the Western Province, assayed at the same time, *i.e.*, some four weeks ago, were each found to contain 4·8 grains of platinum per ton of 2000 lbs.

The possibility of the platinum having come from the rocks appeared to be so remote that it was deemed advisable to test the assay materials in order to ascertain whether they were platiniferous. The hydric sodic carbonate having been proved free from platinum on previous occasions, only the litharge and the borax were examined. In place of possibly platiniferous rocks four assay ton charges of sea-sand were used, and it was then found that when 160 grms. of the litharge previously used were made into a charge with platinum-free borax, a nil result as regards platinum followed, whereas, when 40 grms. of the borax employed in the above assays was coupled with platinum-free litharge, 0·04 of a mgrm. of platinum was invariably obtained as the result of the assay.

In such cases the silver bead from the assay showed the peculiar dull spongy appearance which is characteristic of beads containing over 5 per cent platinum. On dissolving this particular bead in hot concentrated sulphuric acid a few specks of a black powder were left which weighed 0·04 of a mgrm. This powder when dissolved in aqua regia, the solution being evaporated to dryness and the residue taken up with water, gave the platinum reaction with potassium iodide. Lest it should be thought that the platinum could have been derived from the sea-sand an assay was made containing only borax, platinum-free litharge, and sufficient carbon to produce a lead button. In this case, too, the bead produced contained the quantity of platinum already stated.

In order to confirm this result further assays were undertaken, one on 200 grms. of borax and another on 1000 grms. of borax, with the following results:—

200 grms. of borax yielded 0·26 mgrm. of black powder, 1000 grms. of borax yielded 1·42 mgrms. of black powder.

The 0·26 of a mgrm., when converted into chloride and treated with ammonia chloride and alcohol, yielded the characteristic yellow precipitate, which, upon ignition, was converted into spongy platinum, and from this was once more obtained yellow ammonic chloro-platinate.

The 1·42 mgrms., when treated with aqua regia, did not completely dissolve, but left a residue weighing 0·12 of a mgrm. This residue did not appreciably lose weight after eight hours' treatment with boiling aqua regia, and appeared under the lens as greyish metallic spangles, which were unaltered by ignition. Unfortunately there was not sufficient borax available from which to obtain enough of this substance to determine its nature. Having regard, however, to the foregoing tests and the manner in which the residue was obtained, it seems highly probable that it consists of iridium osmide.

The chloride soluble in water was converted into ammonic chloro-platinate.

The results available, therefore, are as follows:—

40 grms. borax used for assaying dolerite gave 0·04 mgrm. platinum.

40 grms. borax used for assaying quartz gave 0·4 mgrm. platinum.

40 grms. borax used with sea-sand in place of rock gave 0·04 mgrm. platinum.

200 grms. borax used with sea-sand in place of rock gave 0·26 mgrm. platinum.

1000 grms. borax used with sea-sand in place of rock gave 1·30 mgrms. platinum and 0·12 mgrm. osmiridium.

It therefore appears certain that the borax contains platinum to the amount of 1·3 parts per million, *i.e.*, 0·00013 per cent.

In casting about for the source of platinum it seemed possible that this impurity might have been introduced by fusing the borax in platinum vessels, assuming such to have been used in its manufacture. In order to test this theory 250 grms. of platinum-free borax was fused in a platinum basin at a red heat for four hours. The melt was allowed to cool, and when removed from the dish 200 grms. of it yielded 0·6 mgrm. platinum. A second 250 grms. was treated in a similar manner, but in this case so as to obviate the possibility of any platinum having been mechanically removed by the hand-fused borax, the latter was poured while still liquid into clean sand. 200 grms. of this yielded 0·46 mgrm. platinum. A third quantity confirmed the result obtained in the second experiment.

It is thus shown that fused borax has a very distinct action on platinum basins.

It may be interesting to note that up to the time that platinum was discovered in the borax ordinary assay borax had been employed. The comparatively large quantities of platinum were found in Merck's "m. g. r. puriss. borax." Even ordinary assay borax is not always entirely free from platinum, traces of the noble metal having recently been found to exist in a sample at present in use in the Government Laboratory.

Under ordinary circumstances the occurrence of such minute traces of platinum in an assay chemical would not

* Read before the Cape Chemical Society, Cape Town, July 17, 1908.

be of more than academic interest, for it would probably not be seriously asserted that rocks containing the infinitesimal traces of the metal which the use of this platiniferous borax would make them appear to contain could pay for working, and as a rule, therefore, time was not wasted in looking for such traces. In view, however, of recent allegations regarding the occurrence of platinum metals in South African rock formations the matter assumes a somewhat different complexion.

The assay balance used in the foregoing experiments turns easily with one-fiftieth mgrm., and with care can be made to indicate one-hundredth mgrm.

TECHNICAL METHOD FOR THE DETERMINATION OF LEAD IN ORES, &c.

By A. H. LOW.

The following scheme is the result of many attempts on my part to improve the methods wherein lead is separated as oxalate and subsequently titrated with permanganate. I have used the method as described below for several months, and have found it more satisfactory for technical work than any other with which I am acquainted. Duplicate assays usually check within one-tenth of 1 per cent. A test of five portions of pure lead, weighed so as to approximately represent 3, 12, 20, 45, and 60 per cent of lead in an ore, and put through the entire process, showed errors of −0·01, −0·06, +0·01, +0·05, and +0·05 per cent respectively, the average error being 0·036 per cent. The permanganate was standardised on about 0·200 grm. of lead.

Chromate-Oxalate Method.—Take 0·5 grm. of ore and treat in a 6-oz. flask by the usual methods to obtain the washed lead sulphate, &c., on a 9 cm. filter. Dissolve the lead sulphate on the filter by stirring it up repeatedly with a jet of hot sodium acetate solution contained in a wash-bottle, receiving the filtrate in the original flask. Prepare the solvent by diluting a cold saturated solution of commercial sodium acetate with an equal bulk of water and adding 40 cc. of 80 per cent glacial acetic acid per litre. To test if the extraction of the lead sulphate is complete, the flask may be replaced by a small beaker, the washing continued, and a little potassium dichromate added to the filtrate. If any lead chromate is produced, the mixture may be added to the clear solution in the flask. Agitate the extract in the flask and heat it if necessary to re-dissolve any separated precipitate. Add 10 cc. of a 5 per cent solution of commercial potassium dichromate, heat to boiling, and boil gently for a few minutes to render the precipitate basic and easily filtered. The change is shown by its becoming reddish yellow in colour. Filter hot, wash out the flask with hot water, and then wash the precipitate only once, simply to clean the upper edge of the filter. Place a wide-necked funnel in the flask, open the filter and spread it against the wall of the funnel, and wash off the lead chromate with a jet of hot oxalic acid solution, using from 25 to 40 cc. Rinse down any adhering chromate in the flask with hot water. The oxalic acid solution consists of a cold saturated solution of commercial acid 1 part, water 3 parts. Heat nearly to boiling in a wash-bottle. To the mixture in the flask add grain alcohol and then boil until the chromic acid is all reduced and the lead converted to oxalate. Remove from the heat, add 30 cc. of cold water and cool thoroughly, best under the tap or in cold water. When cold, filter through an 11 cm. filter, wash out the flask thoroughly with cold water, and then wash filter and precipitate ten times with cold water. Place 5 cc. of strong sulphuric acid in the flask, dilute first with a little cold water and then with hot water to about 125 cc. Add the filter and precipitate and titrate to the usual pink tinge with standard potassium permanganate solution. The solution used for iron will serve, although too strong for the best work. Theoretically, the oxalic acid ($C_2O_4H_2.2H_2O$) value of the permanganate multiplied by 1·642 will give the lead value, but owing to slight losses as sulphate, oxalate, &c., and the fact that the lead oxalate is not perfectly pure, the factor 1·669 will give a closer approximation. On this basis, for 0·5 grm. of ore taken for assay, the solution should contain 1·5185 grms. of potassium permanganate per litre, in order that 1 cc. may equal 1 per cent lead. It is best to standardise on about 0·200 grm. of pure lead dissolved in a little 1 : 2 nitric acid and put through the entire process.

Notes.

The lead oxalate formed in the above process is not pure white, but yellowish, and still contains about 1 per cent of lead chromate. It appears to be sufficiently uniform in its nature to give accurate results.

Instead of dropping filter and precipitate into the flask for titration, as described above, a neater method of procedure is as follows:—Place the flask under the funnel and pour through and over the filter about 75 cc. of hot dilute acid, containing 5 cc. of strong sulphuric acid, and then wash the filter and residue well with hot water, so that the final bulk of the filtrate will be about 125 cc. Titrate the hot liquid as before. This method takes a little longer and gives practically the same results as the simpler way, but it has the advantage of a permanent end-point, there being no organic matter present to slowly decolorise the pink tinge.

Calcium does not interfere with the method, nor does antimony. Bismuth in small amounts is without material influence. Ten per cent of bismuth added to a mixture containing about 23 per cent of lead raised the result 0·36 per cent, most of the bismuth being removed as sulphate and chromate.—*Journal of the American Chemical Society*, xxx., p. 587.

A SEPARATION OF IRON FROM MANGANESE.

By RICHARD B. MOORE and IVY MILLER.

If to a solution of ferric chloride containing free hydrochloric acid, pyridine, in slight excess, be added, the iron is completely precipitated as hydroxide. Aluminium, chromium, and zinc, under such conditions, are not completely precipitated, while manganese, nickel, and cobalt remain in solution. This method can therefore be used to separate iron from the last three metals. The present paper deals only with the separation of manganese from iron by means of pyridine.

When pyridine is added to a neutral solution of a manganous salt no precipitate is obtained. On warming, the solution slowly oxidises and the manganese begins to come down. The rate at which oxidation takes place, however, is about one-third as fast as the rate at which a manganous solution oxidises when treated with ammonium chloride and ammonium hydroxide under similar conditions. If the manganous solution is made acid with a little hydrochloric acid before addition of the pyridine to slight excess, the solution may then be heated for ten minutes without oxidation. Consequently, pyridine possesses considerable advantages over ammonium hydroxide in the separation under question, especially when a large amount of manganese is present. Practically, its only disadvantage is its cost. A very fair separation can be made with one precipitation.

On washing ferric hydroxide precipitated by pyridine, no iron at first appears in the filtrate. As the excess of pyridine is washed out of the precipitate, however, colloidal iron passes through the filter. On washing the precipitate with pyridine water (1 : 500) the iron is completely retained by the filter.

In the following experiments Merck's pyridine, "Medicinal grade," was used. It was re-distilled before using.

Pure iron wire was dissolved in 10 cc. of 4/N hydrochloric acid. A few drops of concentrated nitric acid were

added and the solution warmed to oxidise the iron. It was then diluted to 100 cc., warmed, and pyridine added from a burette. The precipitate was washed with pyridine water (1 : 500) :—

No.	Iron taken. Grm.	Iron found. Grm.
1.	0·0806	0·0807
2.	0·0823	0·0824
3.	0·0798	0·0810

In No. 1, the pyridine was added until the iron was just precipitated. In No. 2 there was an excess of 0·5 cc. of pyridine, and in No. 3 an excess of 1 cc.

Solutions of manganous chloride were then mixed in different proportions with ferric chloride. The amount of free acid was the same as was taken above. The precipitation takes place equally well in the cold or after gently warming. In the following series the manganese in the filtrate was not estimated, the object being to see what kind of a separation was made by a single precipitation. One-half a cubic centimetre in excess of pyridine was used in each case :—

No.	Iron taken. Grm.	Iron found. Grm.	Manganese present. Grm.
1.	0·0797	0·0798	0·1405
2.	0·0790	0·0792	0·0857
3.	0·0791	0·0792	0·0719
4.	0·0798	0·0802	0·0610

In the next series both the iron and manganese were estimated in order to see what effect the presence of the pyridine has on the precipitation of the manganese. The manganese cannot be completely precipitated by addition of bromine water to the hot solution. In addition, the formation of pyridine bromide interferes. In No. 1 the manganese was estimated in the usual manner by potassium carbonate. In Nos. 2, 3, and 4, chemically pure sodium hydroxide was used instead of the carbonate, the precipitation being made in porcelain basins :—

	Iron.		Manganese.	
No.	Taken. Grm.	Found. Grm.	Taken. Grm.	Found. Grm.
1.	0·0858	0·0862	0·0698	0·0691
2.	0·1136	0·1133	0·0667	0·0666
3.	0·1162	0·1155	0·2289	0·2285
4.	0·1268	0·1264	0·1145	0·1140

The conditions under which aluminium can be separated from manganese and aluminium and iron from beryllium by means of pyridine are under investigation.—*Journal of the American Chemical Society*, xxx., p. 593.

ON THE ESTIMATION OF CERIUM IN THE PRESENCE OF THE OTHER RARE EARTHS BY THE ACTION OF POTASSIUM FERRICYANIDE.

By PHILIP E. BROWNING and HOWARD E. PALMER.

The work to be described was undertaken to determine how completely the oxidation of cerium from the cerous to the ceric condition may be effected by potassium ferricyanide in alkaline solution, and how completely the measure of the oxidation can be registered in the amount of potassium ferrocyanide formed, according to the following equation :—

$$2K_3FeC_6N_6 + Ce_2O_3 + 2KOH = 2K_4FeC_6N_6 + H_2O + 2CeO_2.$$

For this work a solution of pure cerous sulphate was made and standardised by precipitating measured and weighed portions with a definite amount of a standard solution of sodium oxalate, filtering, igniting the cerium oxalate, and weighing the ceric oxide obtained. As a check on this method, the excess of the sodium oxalate over the amount used for the precipitate was determined in the filtrate by titration with potassium permanganate, and this amount was subtracted from the whole amount of sodium oxalate used. From this result the cerium present can be readily estimated, the amount of sodium oxalate used in the precipitation being known (Browning and Lynch, *Journ. Am. Sci.*, 1899, [4], viii., 457).

The ferricyanide solution used was made by dissolving 2 grms. of carefully selected crystals of potassium ferricyanide in 100 cc. of water. About 20 cc. of this solution were used in each determination.

The procedure was as follows:—To measured and weighed portions of the cerous sulphate solution 20 cc. of the ferricyanide solution were added, and potassium hydroxide in solution to complete precipitation.

The precipitated hydroxide was filtered off, and the filtrate and washings, amounting in volume to from 200 cc. to 250 cc., after being made distinctly acid with dilute sulphuric acid, were titrated with a standard solution of potassium permanganate until the presence of the permanganate colour showed the oxidation of the ferrocyanide to the ferricyanide, according to the equation—

$$5K_4FeC_6N_6 + KMnO_4 + 4H_2SO_4 = 5K_3FeC_6N_6 + 3K_2SO_4 + MnSO_4 + 4H_2O$$

(Sutton's "Volumetric Analysis," Ninth Edition, p. 209). By this equation and the preceding one the amount of cerium present can be readily calculated.

Each day before the ferricyanide was used a portion of 20 cc. of the solution was acidified and titrated with the permanganate to colour, and the amount necessary, generally from one to three drops, was subtracted from the amount of the permanganate used in the actual determination. It is of interest to note that ferricyanide solutions kept in clear glass bottles for a week or more showed variations of only a drop or two in the amount of permanganate taken up.

TABLE I.

	Ce taken, calculated as Ce_2O_3. Grm.	Ce found, calculated as Ce_2O_3. Grm.	Error. Grm.
1.	0·1834	0·1819	−0·0015
2.	0·1376	0·1380	+0·0004
3.	0·1834	0·1829	−0·0005
4.	0·1834	0·1829	−0·0005
5.	0·1834	0·1834	—
6.	0·1376	0·1385	+0·0009
7.	0·1376	0·1371	−0·0005
8.	0·1376	0·1374	−0·0002
9.	0·1376	0·1380	+0·0004
10.	0·1834	0·1824	−0·0010
11.	0·1326	0·1335	+0·0009
12.	0·1326	0·1328	+0·0002

TABLE II.

	Ce taken, calculated as Ce_2O_3. Grm.	Ce found, calculated as Ce_2O_3. Grm.	Error. Grm.	Other rare earths present, calculated as oxides. Grm.
1.	0·1328	0·1335	+0·0007	0·1 ThO_2
2.	0·1327	0·1322	−0·0005	0·1 "
3.	0·0266	0·0275	+0·0009	0·1 "
4.	0·0267	0·0272	+0·0005	0·1 "
5.	0·1324	0·1326	+0·0002	0·1 Y_2O_3
6.	0·1326	0·1323	−0·0003	0·1 "
7.	0·0266	0·0264	−0·0002	0·1 "
8.	0·0264	0·0271	+0·0005	0·1 "
9.	0·1376	0·1370	−0·0006	0·15 $La_2O_3 + Di_2O_3$
10.	0·1101	0·1091	−0·0010	0·15 "
11.	0·1324	0·1332	+0·0008	0·03 ZrO_2 "

All the various operations in this process were carried on without warming the solution. The filtrations and washings were generally made under gentle pressure, and required on an average not more than fifteen to thirty minutes. In Table I. the results obtained with cerium present alone are given.

Solutions of the salts of the other rare earths, containing about 0·1 grm., were treated by the same method, and the failure to obtain evidence, by the permanganate, of the formation of potassium ferrocyanide, showed that there is no oxidation of these salts by the ferricyanide.

In Table II. the results obtained by estimating cerium according to this method in the presence of the other rare earths are given.

This method presents no difficulties in manipulation, and is especially adapted to the rapid estimation of cerium in rare earth mixtures.—*American Journal of Science*, xxvi., p. 83.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 94).

The Iron Group.

Procedure 61.—Transfer the Na_2O_2 precipitate (P. 52) to a casserole together with the filter if necessary, add 5—30 cc. HCl (1·12), boil gently till the precipitate is dissolved, filter to remove the paper, and evaporate the filtrate to 1 or 2 cc. To decompose the HCl add about 5 cc. HNO_3 (1·42), and boil as long as oxides of nitrogen are given off. Add 5—20 cc. HNO_3 (1·42), heat to boiling, add about 0·5 grm. of solid $KClO_3$ and boil gently, adding more $KClO_3$ in small portions if a large precipitate forms. (Dark brown or black precipitate, presence of *manganese*). Boil gently for a minute or two, and filter through an asbestos filter, made by pouring a suspension of washed asbestos over a compact wad of glass-wool in a glass funnel. (See Note 4). Heat the filtrate to boiling, add more $KClO_3$, boil, and filter through the same filter if more of the precipitate separates. Wash two or three times with HNO_3 (1·42) which has previously been freed from the oxides of nitrogen by warming with a little $KClO_3$. Evaporate the filtrate to about 5 cc., but not further, dilute to 20 or 30 cc., and filter the solution if it is turbid. (Precipitate, P. 62; filtrate, P. 63).

Notes.

1. HCl is used for dissolving the Na_2O_2 precipitate rather than HNO_3. Pure concentrated HNO_3 does not dissolve hydrated MnO_2 except in the presence of filter-paper, whereby the HNO_3 is reduced to lower oxides; the action is more rapid with HCl (for the MnO_2 is thereby quickly reduced to manganous chloride with evolution of chlorine).

2. By $HClO_3$ in HNO_3 solution (but not by HNO_3 alone) manganous salts are rapidly oxidised to hydrated MnO_2 with formation of chlorine dioxide (ClO_2), which escapes as a yellow gas. The HCl must previously be completely removed by evaporation and boiling with HNO_3, since the oxides of nitrogen resulting from its action on the HNO_3 would continuously dissolve the precipitate.

3. The separation of manganese in this way from the other metals of this group is entirely satisfactory with the following exceptions. A small quantity of iron (up to 1 mgrm.) may be completely carried down with a large quantity (500 mgrms.) of manganese. The same is true in a much higher degree of titanium, of which even 50 mgrms. may be entirely precipitated with 500 mgrms. of manganese. Much zirconium is also carried down, but never quite completely. Provision is therefore made (in *P. 62*a*) for the detection of these three elements in the precipitate (if it is large) as well as in the filtrate. If it is not thought necessary to test for zirconium in the precipitate, the certain detection of titanium can be much more quickly accomplished just before precipitating the manganese, by diluting the HNO_3 solution, from which the HCl has been removed, with once or twice its volume of water, and adding 3 cc. of 3 per cent H_2O_2 solution. An orange-yellow or orange-red colour shows the presence of titanium. (See *P. 65*b*, N. 1). In the absence of HCl iron does not interfere with this test, nor does even a large quantity of nickel or cobalt prevent a distinct change of colour from being seen.

4. In filtering the MnO_2 a cylindrical glass funnel with a small delivery tube is usually employed in quantitative analysis. An ordinary conical funnel is, however, satisfactory, provided the wad of glass-wool is made compact and enough asbestos is used. Filtration will, to be sure, be slow if the glass-wool is packed very tightly or if the asbestos mat is very thick, but in that case suction may be applied.

5. On evaporating the HNO_3 filtrate, titanium and zirconium oxides may separate out, especially if the solution be evaporated almost to dryness. For this reason the solution is evaporated only to about 5 cc., but even then a small white precipitate will sometimes be obtained when these elements are present in large amount.

Procedure 62.—Transfer the whole of the $HClO_3$ precipitate (P. 61) if it is small (containing less than 5 mgrms. Mn), or 5—10 mgrms. of it if it is large, to a casserole; add 1 or 2 grms. solid PbO_2 and about 10 cc. HNO_3 (1·20); boil for about two minutes in a casserole covered with a watch-glass; pour the mixture into a test-tube, and allow the PbO_2 to settle. (Violet-red solution, presence of *manganese*).

If the precipitate is large, dissolve the remainder in hot HCl (1·12) in a casserole, or by pouring a 10—15 cc. portion repeatedly through the filter, and boil the solution to expel chlorine. To one-tenth of this solution add 5 cc. KCNS solution. (Deep red colour, presence of *iron*). Treat the remainder of the HCl solution by *P. 62*a* to recover any titanium and zirconium that may be present.

Notes.

1. This confirmatory test for manganese is usually superfluous since the precipitation of MnO_2 by $HClO_3$ is highly characteristic. In order that the PbO_2 test may be satisfactory, the HNO_3 used must be fairly concentrated and the boiling continued for two or three minutes.

Procedure 62*a*.—To the HCl solution (P. 62), without evaporating it, add NH_4OH (0·96) until the mixture is barely alkaline, avoiding the addition of more than two or three drops in excess; heat nearly to boiling for two or three minutes, filter at once, and wash with hot water. Reject the filtrate. Dissolve the precipitate in a little hot HCl (1·12) (without diluting it with water), reserve it, and unite it with the main HCl solution to be treated in *P. 65*a*.

Notes.

1. In order to avoid as far as possible the precipitation of manganese by NH_4OH, the OH^- concentration is kept small by avoiding an excess of NH_4OH and by having a large quantity of ammonium salt present. Under these conditions the oxidation by the air to the manganic state is slow. If, however, much NH_4OH be added, oxidation takes place rapidly and much

* From the *Journal of the American Chemical Society*, xxx., No. 4.

manganese may be precipitated as $Mn(OH)_3$, yielding a brown precipitate. Even at best a little manganese will come down, but a moderate amount does not interfere with the subsequent tests for titanium and zirconium.

Procedure 63.—Add about one-tenth of the HNO_3 solution (P. 61) to three or four times its volume of ammonium molybdate reagent, and heat to 60—70°. (Yellow, finely crystalline precipitate, presence of *phosphate*). If there is no precipitate, or only a very small one, treat the remainder of the HNO_3 solution by P. 64; otherwise by P. 65.

Notes.

1. Phosphate is tested for at this point because a different treatment is necessary when it is present in significant amount, in order to separate from it alkaline-earth elements and to provide for their detection. When phosphate is not present, iron, thallium, titanium, and zirconium can be separated from nickel, cobalt, and the alkaline-earth elements by NH_4OH (as in P. 64); but, when considerable phosphate is present, the alkaline-earth elements would be partly or wholly precipitated in combination with it. (See P. 51, N. 6).

2. In order that the phosphate test may be delicate and may appear immediately, a large proportion of the molybdate reagent must be used and the solution must be warmed. The precipitate of ammonium phospho-molybdate is of complicated and somewhat variable composition; it contains ammonium phosphate and molybdic acid, approximately in the proportion $(NH_4)_3PO_4 : 12MoO_3$.

Procedure 64.—If phosphate is absent, make the HNO_3 solution (P. 62) strongly alkaline with NH_4OH (0·96) using an excess of 3—5 cc. (Dark red precipitate, presence of *iron*). Filter, and wash the precipitate, using suction if the precipitate is large, and sucking it as dry as possible. Treat the filtrate by P. 66.

Dissolve the precipitate in HCl (1·12), warming if necessary, taking care not to dilute the acid by wash water. To about one-tenth of the solution add 5 cc. KCNS solution. (Dark red colour, presence of *iron*). *Treat the remainder of the solution by *P. 65*a*.

Notes.

1. If titanium and zirconium are to be tested for, it is important to dissolve the NH_4OH precipitate in HCl of a specific gravity 1·12 (24 per cent HCl) and to avoid dilution, for the separation in *P. 65*a* depends on the concentration of the HCl.

2. The red colour obtained on adding KCNS is due to the formation of un-ionised ferric thiocyanate, $Fe(CNS)_3$. This test may be made in the presence of much HCl, for the acid HCNS is also a highly dissociated acid, which is therefore not displaced from its salt. Much HNO_3 must not, however, be present; for, by its action on KCNS, NO_2 may be formed, and this also gives a deep red colour with KCNS. This test for iron is an extremely delicate one; and if only a faint colour is obtained, the acids used in the process must be tested for iron.

(To be continued).

MISCELLANEOUS.

Preparation of a Solution for Making Standard Solutions of Sodium Hydroxide.—H. W. Cowles, Jun. —Prepare an approximately 50 per cent solution of sodium hydroxide by dissolving 50 grms. of ordinary chemically pure caustic soda sticks in 50 cc. of distilled water and leave tightly stoppered over night. The major portion of the impurities is insoluble in such a concentrated solution. The next day the proper amount of the clear supernatant solution is weighed or measured out, as most convenient, and diluted to the mark with freshly boiled distilled water. Theoretically 8 grms. or 5·25 cc. are required per litre of N/10 solution, but owing to the impurities which have settled out it is safer to use 8·5 grms. or 5·75 cc. per litre, and then standardise against N/10 acid. With such a standard solution, made up as directed and kept in an automatic burette with a potash bulb to purify the air that enters the apparatus, the strength is maintained indefinitely, while one drop of N/10 H_2SO_4 is more than sufficient to render 25 cc. of the solution, already neutralised to phenol-phthalein, acid to methyl orange, showing that it is practically completely free from carbonates, &c. In proof of this, barium hydroxide gives no precipitate when added to 25 cc. of the solution. The remainder of the strong caustic soda solution, if kept tightly stoppered, will furnish material for the preparation of future supplies when needed.—*Journal of the American Chemical Society*, xxx., No. 7.

The Iron and Steel Institute.—President, Sir Hugh Bell, Bart.—In accordance with previous announcements, the Autumn Meeting of the Iron and Steel Institute will be held at Middlesbrough on Monday, Tuesday, Wednesday, Thursday, and Friday, September 28th, 29th, and 30th, October 1st and 2nd, 1908. An influential Reception Committee has been formed, with the Worshipful the Mayor of Middlesbrough (Lieut.-Colonel T. G. Poole, V.D.) as Chairman, and Mr. John H. Amos (Queen's Terrace, Middlesbrough) as Hon. Secretary. The following papers have been offered for reading:—

"Scientific Control of Fuel Supply." By Prof. H. E. Armstrong, Ph.D., LL.D., F.R.S. (London).
"Metallurgy at the Franco-British Exhibition." By H. Bauerman, Hon. Member.
"Gas Producer Practice." By Prof. W. A. Bone, D.Sc., F.R.S. (Leeds), and R. V. Wheeler, D.Sc. (Normanton).
"Constitution of Carbon Steels." By Prof. E. D. Campbell (Ann Arbor, Michigan, U.S.A.).
"Freezing-point of Iron." By Prof. H. C. H. Carpenter, M.A., Ph.D. (Manchester).
"Production of Finished Iron Sheets and Tubes in One Operation." By S. O. Cowper-Coles (London).
"Chemical Control of the Basic Open-Hearth Process." By Alfred Harrison (Warrington), and R. V. Wheeler, D.Sc. (Normanton).
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"Electric Power Supply." By Charles H. Merz (London).
"Future Development of the Metal Mixer." By A. E. Pratt, Assoc.R.S.M. (London).
"Test for Ascertaining the Relative Wearing Properties of Steel Rails." By E. H. Saniter (Rotherham).
"Workshop Microscope." By J. E. Stead, F.R.S. (Member of Council).
"Analysis and Synthesis in the Foundry." By J. E. Stead, F.R.S. (Member of Council), and T. Westgarth (Middlesbrough).

Members who intend to take part in the discussion will, on notifying such intention to the Secretary, have copies of the papers forwarded to them a week in advance, as far as that may be possible.

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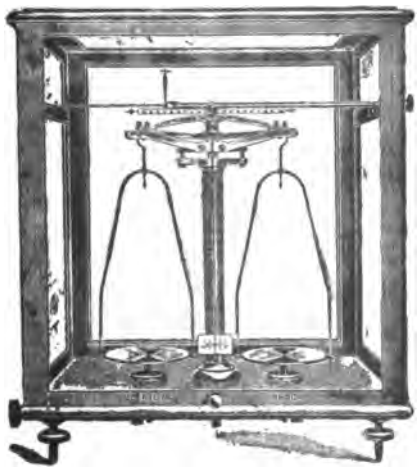

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FRANCIS DARWIN, M.A., Ph.D., LL.D., F.R.S.

BEFORE entering on the subject of my Address, I may be allowed to refer to the loss which the British Association has sustained in the death of Lord Kelvin. He joined the Association in 1847, and had been for more than fifty years a familiar figure at our meetings. This is not the occasion to speak of his work in the world or of what he was to his friends, but rather of his influence on those who were personally unknown to him. It seems to me characteristic of him that something of his vigour and of his personal charm was felt far beyond the circle of his intimate associates, and many men and women who never exchanged a word with Lord Kelvin, and are in outer darkness as to his researches, will miss his genial presence and feel themselves the poorer to-day. By the death of Sir John Evans the Association is deprived of another faithful friend. He presided at Toronto in 1897, and since he joined the Association in 1861 had been a regular attendant at our meetings. The absence of his cheerful personality and the loss of his wise counsels will be widely felt.

May I be permitted one other digression before I come to my subject? There has not been a Botanical President of the British Association since the Norwich meeting forty years ago, when Sir Joseph Hooker was in the chair, and in "eloquent and felicitous words" (to quote my father's letter) spoke in defence of the doctrine of evolution. I am sure that every member of this Association will be glad to be reminded that Sir Joseph Hooker is, happily, still working at the subject that his lifelong labours have so greatly advanced, and of which he has long been recognised as the honoured chief and leader.

You will perhaps expect me to give a retrospect of the progress of evolution during the fifty years that have elapsed since July 1, 1858, when the doctrine of the origin of species by means of natural selection was made known to the world in the words of Mr. Darwin and Mr. Wallace. This would be a gigantic task, for which I am quite unfitted. It seems to me, moreover, that the first duty of your President is to speak on matters to which his own researches have contributed. My work—such as it is—deals with the movements of plants, and it is with this subject that I shall begin. I want to give you a general idea of how the changes going on in the environment act as stimuli and compel plants to execute certain movements. Then I shall show that what is true of those temporary changes of shape we describe as movements is also true of the permanent alterations known as morphological.

I shall insist that, if the study of movement includes the problem of stimulus and reaction, morphological change must be investigated from the same point of view. In fact, that these two departments of inquiry must be classed together, and this, as we shall see, has some important results—namely, that the dim beginnings of habit or unconscious memory that we find in the movements of plants and animals must find a place in morphology; and inasmuch as a striking instance of correlated morphological changes is to be found in the development of the adult from the ovum, I shall take this ontogenetic series and attempt to show you that here also something equivalent to memory or habit reigns.

Many attempts have been made to connect in this way the phenomena of memory and inheritance, and I shall ask you to listen to one more such attempt, even though I am forced to appear as a champion of what some of you consider a lost cause—the doctrine of the inheritance of acquired characters.

Movement.

In his book on "The Power of Movement in Plants," 1880, p. 571, my father wrote that "it is impossible not to be struck with the resemblance between the foregoing movements of plants and many of the actions performed unconsciously by the lower animals." In the previous year Sachs ("Arbeiten, 1879, ii., p. 282) had in like manner called attention to the essential resemblance between the irritability of plants and animals. I give these statements first because of their simplicity and directness; but it must not be forgotten that before this Pfeffer had begun to lay down the principles of what is now known as *Reizphysiologie*, or the physiology of stimulus, for which he and his pupils have done so much ("Osmotische Untersuchungen," 1877, p. 202).

The words of Darwin which I have quoted afford an example of the way in which science returns to the obvious. Here we find revived, in a rational form, the point of view of the child or of the writer of fairy stories. We do not go so far as the child; we know that flowers do not talk or walk; but the fact that plants must be classed with animals as regards their manner of reaction to stimuli has now become almost a commonplace of physiology. And inasmuch as we ourselves are animals, this conception gives us a certain insight into the reactions of plants which we should not otherwise possess. This is, I allow, a very dangerous tendency, leading to anthropomorphism, one of the seven deadly sins of science. Nevertheless, it is one that must be used unless the great mass of knowledge accumulated by psychologists is to be forbidden ground to the physiologist.

Jennings has admirably expressed the point of view from which we ought to deal with the behaviour of the simpler organisms ("The Behaviour of the Lower Organisms," 1904, p. 124). He points out that we must study their movements in a strictly objective manner: that the same point of view must be applied to man, and that any resemblance between the two sets of phenomena are not only an allowable but a necessary aid to research.

What, then, are the essential characters of stimuli and of the reactions which they call forth in living organisms? Pfeffer has stated this in the most objective way. An organism is a machine which can be set going by touching a spring or trigger of some kind; a machine in which energy can be set free by some kind of releasing mechanism. Here we have a model of at least some of the features of reaction to stimulation.

The energy of the cause is generally out of all proportion to the effect, *i.e.*, a small stimulus produces a big reaction. The specific character of the result depends on the structure of the machine rather than on the character of the stimulus. The trigger of a gun may be pulled in a variety of different ways without affecting the character of the explosion. Just in the same way a plant may be made to curve by altering its angle to the vertical, by lateral illumination, by chemical agency, and so forth; the curvature is of the same nature in all cases, the release-action differs. One of those chains of wooden bricks in which each knocks over the next may be set in action by a touch, by throwing a ball, by an erring dog, in short by anything that upsets the equilibrium of brick No. 1; but the really important part of the game, the way in which the wave of falling bricks passes like a prairie fire round a group of Noah's Ark animals, or by a bridge over its own dead body and returns to the starting-point, &c.—these are the result of the magnificent structure of the thing as a whole, and the upset of brick No. 1 seems a small thing in comparison.

For myself I see no reason why the term *stimulus* should not be used in relation to the action of mechanisms in general; but by a convention which it is well to respect

stimulation is confined to the protoplasmic machinery of living organisms.

The want of proportion between the stimulus and the reply, or, as it has been expressed, the unexpectedness of the result of a given stimulus, is a striking feature in the phenomena of reaction. That this should be so need not surprise us. We can, as a rule, only know the stimulus and the response, while the intermediate processes of the mechanism are hidden in the secret life of protoplasm. We might, however, have guessed that big changes would result from small stimuli, since it is clear that the success of an organism in the world must depend partly at least on its being highly sensitive to changes in its surroundings. This is the adaptive side of the fundamental fact that living protoplasm is a highly unstable body. Here I may say one word about the adaptation as treated in the "Origin of Species." It is the present fashion to minimise or deny altogether the importance of natural selection. I do not propose to enter into this subject; I am convinced that the inherent strength of the doctrine will insure its final victory over the present anti-Darwinian stream of criticism. From the Darwinian point of view it would be a remarkable fact if the reactions of organisms to natural stimuli were not adaptive. That they should be so, as they undoubtedly are, is not surprising. But just now I only call attention to the adaptive character of reactions from a descriptive point of view.

Hitherto I have implied the existence of a general character in stimulation without actually naming it; I mean the indirectness of the result. This is the point of view of Dutrochet, who in 1824 said that the environment suggests but does not directly cause the reaction. It is not easy to make clear in a few words the conception of indirectness. Pfeffer ("Physiology," Engl. edit., i., p. 11) employs the word *induction*, and holds that external stimuli act by producing internal change, such changes being the link between stimulus and reaction. It may seem, at first sight, that we do not gain much by this supposition; but since these changes may be more or less enduring, we gain at least the conception of *after effect* as a quality of stimulation. What are known as *spontaneous* actions must be considered as due to internal changes of unknown origin.

It may be said that in speaking of the "indirectness" of the response to stimuli we are merely expressing in other words the conception of release-action; that the explosion of a machine is an indirect reply to the touch on the trigger. This is doubtless true, but we possibly lose something if we attempt to compress the whole problem into the truism that the organism behaves as it does because it has a certain structure. The quality of indirectness is far more characteristic of an organism than of a machine, and to keep it in mind is more illuminating than a slavish adherence to the analogy of a machine. The reaction of an organism depends on its past history; but, it may be answered, this is also true of a machine the action of which depends on how it was made, and in a less degree on the treatment it has received during use. But in living things this last feature in behaviour is far more striking, and in the higher organisms past experience is all-important in deciding the response to stimulus. The organism is a plastic machine profoundly affected in structure by its own action, and the unknown process intervening between stimulus and reaction (on which the indirectness of the response depends) must have the fullest value allowed it as a characteristic of living creatures.

For the zoological side of biology a view similar to that of Pfeffer has been clearly stated by Jennings in his admirable studies on the behaviour of infusoria, rotifers, &c. ("Contributions to the Study of the Behaviour of the Lower Organisms," Carnegie Institution, 1904, p. 111). He advances strong arguments against the theories of Loeb and others, according to which the stimulus acts directly on the organs of movement; a point of view which was formerly held by botanists, but has since given place to the conception of the stimulation acting on the organism as a whole. Unfortunately for botanists these movements are by the zoologists called *tropisms*, and are thus liable to be confused with the geotropism, heliotropism, &c., of plants; to these movements, which are not considered by botanists to be due to direct action of stimuli, Loeb's assumptions do not seem to be applicable.

Jennings's position is that we must take into consideration what he calls "physiological state, *i.e.*, 'the varying internal physiological conditions of the organisms, as distinguished from permanent anatomical conditions.'" Though he does not claim novelty for his view, I am not aware that it has ever been so well stated. External stimuli are supposed to act by altering this physiological state; that is, the organism is temporarily transformed into what, judged by its reactions, is practically a different creature.

This may be illustrated by the behaviour of *Stentor*, one of the fixed infusoria (Jennings, "Behaviour of the Lower Organisms," 1906, p. 170). If a fine jet of water is directed against the disc of the creature, it contracts "like a flash" into its tube. In about half a minute it expands again, and the cilia resume their activity. Now we cause the current to act again upon the disc. This time the *Stentor* does not contract. which proves that the animal has been in some way changed by the first stimulus. This is a simple example of "physiological state." When the *Stentor* was at rest, before it received the first current of water, it was in state 1, the stimulus changed state 1 into state 2, to which contraction is the reaction. When again stimulated it passed into state 3, which does not produce contraction.

We cannot prove that the contraction which occurred when the *Stentor* was first stimulated was due to a change of state. But it is a fair deduction from the result of the whole experiment, for after the original reaction the creature is undoubtedly in a changed state, since it no longer reacts in the same way to a repetition of the original stimulus.

Jennings points out that, as in the case of plants, spontaneous acts are brought about when the physiological state is changed by unknown causes, whereas in other cases we can point to an external agency by which the same result is effected.

Morphological Changes.

Let us pass on to the consideration of the permanent or morphological changes, and the stimuli by which they are produced, a subject to which, in recent years, many workers have devoted themselves. I need only mention the names of Vöchting, Goebel, and Klebs among botanists, and those of Loeb, Herbst, and Driesch among zoologists, to remind you of the type of research to which I refer.

These morphological alterations produced by changes in environment have been brought under the rubric of reaction to stimulation, and must be considered as essentially similar to the class of temporary movements of which I have spoken.

The very first stage in development may be determined by a purely external stimulus. Thus the position of the first cell-wall in the developing spore of *Equisetum*, is determined by the direction of incident light (Stahl, *Ber. d. Bot. Ges.*, 1885, p. 334). In the same way the direction of light settles the plane of symmetry of *Marchantia* as it develops from the gemma (Pfeffer, in Sachs' "Arbeiten," i., p. 92). But the more interesting cases are those where the presence or absence of a stimulus makes an elaborate structural difference in the organism. Thus, as Stahl has shown (*Jenaische Zeit.*, 1883, p. 162), beech leaves developed in the deep shade of the middle of the tree are so different in structure from leaves grown in full sunlight that they would unhesitatingly be described as belonging to different species. Another well-known case is the development of the scale-leaves on the rhizome of *Circæa* into the foliage leaves under the action of light (Goebel in *Bot. Zeitung*, 1880).

The power which the experimenter has over the lower plants is shown by Klebs, who kept *Saprolegnia mixta*, a fungus found on dead flies, in uninterrupted vegetative growth for six years; while by removing a fragment of the plant and cultivating it in other conditions the reproductive organs could at any time be made to appear ("Willkürliche Entwick.," p. 27).

Chlamydomonas media, a unicellular green alga, when grown in a 0·4 per cent nutrient solution, continues to increase by simple division, but conjugating gametes are formed in a few days if the plant is placed in pure water, and kept in bright light (Klebs, "Bedingungen," 1896, p. 430). Numberless other cases could be given of the regulation of form in the lower organisms. Thus, *Sporodinia* grown on peptone-gelatine produces sporangiferous hypha, but on sugar zygotes are formed. Again, *Protosiphon botryoides*, if grown on damp clay, can most readily be made to produce spores by transference to water either in light or in darkness. But for the same plant cultivated in Knop's solution the end can best be obtained by placing the culture in the dark (*Biol. Centralbl.*, 1904, pp. 451—3). Still these instances of the regulation of reproduction are not so interesting from our point of view as some of Klebs' later results (*Jahrb. f. wiss. Bot.*, 1906, xlii., p. 162). Thus he has shown that the colour of the flower of *Campanula trachelium* can be changed from blue to white and back again to blue by varying the conditions under which the plant is cultivated. Again, with *Sempervivum* he has been able to produce striking results —*e.g.*, the formation of apetalous flowers with one instead of two rows of stamens (*Abhandl. Naturforsch. Ges. zu Halle*, 1906, xxv., pp. 31, 34, &c.). Diminution in the number of stamens is a common occurrence in his experimental plants, and absolute loss of these organs also occurs. Many other abnormalities were induced, both in the stamens and in other parts of the flowers.

There is nothing new in the character of these facts*; what has been brought to light (principally by the work of Klebs) is the *degree* to which ontogeny is controllable. We are so much in the habit of thinking of the stable element in ontogeny that the work of Klebs strikes us with something of a shock. Most people would allow that change of form is ultimately referable to changed conditions, but many of us were not prepared to learn the great importance of external stimuli in ontogeny.

Klebs begins by assuming that every species has a definite *specific structure*, which he compares to chemical character. Just as a substance such as sulphur may assume different forms under different treatment, so he assumes that the specific structure of a plant has certain potentialities which may be brought to light by appropriate stimuli. He divides the agencies affecting the structure into external and internal conditions, the external being supposed to act by causing alterations in the internal conditions.

It will be seen that the scheme is broadly the same as that of Pfeffer for the case of the movement and other temporary reactions. The internal conditions of Klebs correspond also to the "physiological state" of Jennings.

From what has gone before, it will be seen that the current conception of stimulust is practically identical whether we look at the phenomena of movement or those of structure. If this is allowable—and the weight of evidence is strongly in its favour—a conclusion of some interest follows.

If we reconsider what I have called the indirectness of stimulation, we shall see that it has a wider bearing than is at first obvious. The "internal condition" or "physiological state" is a factor in the regulation of the organism's action, and it is a factor which owes its character to external agencies which may no longer exist.

The fact that stimuli are not momentary in effect, but leave a trace of themselves on the organism is, in fact, the physical basis of the phenomena grouped under memory in its widest sense as indicating that action is regulated by past experience. Jennings remarks (*loc. cit.*, 1904, p. 124): —"In the higher animals, and especially in man, the essential features in behaviour depend very largely on the history of the individual; in other words, upon the present physiological condition of the individual, as determined by the stimuli it has received and the reactions it has performed. But in this respect the higher animals do not differ in principle, but only in degree, from the lower organisms . . . ." I venture to believe that this is true of plants as well of animals, and that it is further broadly true not only of physiological behaviour, but of the changes that are classed as morphological.

Semon in his interesting book, "Die Mneme,"* has used the word *Engram* for the trace or record of a stimulus left on the organism. In this sense we may say that the internal conditions of Pfeffer, the physiological states of Jennings, and the internal conditions of Klebs are, broadly speaking, *Engrams*. The authors of these theories may perhaps object to this sweeping statement, but I venture to think it is broadly true.

The fact that in some cases we recognise the chemical or physical character of the internal conditions does not by any means prevent our ascribing a *mnemic* memory-like character to them, since they remain causal agencies built up by external conditions which have, or may have, ceased to exist. Memory will be none the less memory when we know something of the chemistry and physics of its neutral concomitant.

Habit illustrated by Movement.

In order to make my meaning plain as to the existence of a *mnemic* factor in the life of plants, I shall for the moment leave the morphological side of life and give an instance of habitual movement.

Sleeping plants are those in which the leaves assume at night a position markedly different from that shown by day. Thus the leaflets of the scarlet-runner (*Phaseolus*) are more or less horizontal by day and sink down at night. This change of position is known to be produced by the alternation of day and night. But this statement by no means exhausts the interest of the phenomenon. A sensitive photographic plate behaves differently in light and darkness; and so does a radiometer, which spins by day and rests at night.

If a sleeping-plant is placed in a dark room after it has gone to sleep at night, it will be found next morning in the light-position, and will again assume the nocturnal position as evening comes on. We have, in fact, what seems to be

* See the great collection of facts illustrating the "direct and definite action of the external conditions of life" in "Variation of Animals and Plants," ii., 271).

† With regard to the terminology of stimulation, I believe that it would greatly simplify matters if our classification of casual conditions could be based on the relation of the nucleus to the rest of the cell. But our knowledge does not at present allow of more than a tentative statement of such a scheme. It is now widely believed that the nucleus is the bearer of the qualities transmitted from generation to generation, and the regulator of ontogeny. May we not therefore consider it probable that the nucleus plays in the cell the part of a central nervous system? In plants there is evidence that the ectoplasm is the sensitive region, and, in fact, plays the part of the cell's sense-organ. The change that occurs in the growth of a cell, as a response to stimulus, would on this scheme be a reflex action dependent for its character on the structure of the nucleus. The "indirectness" of stimulation would then depend on the reception by the nucleus of the excitation set up in the ectoplasm, and the secondary excitation reflected from the nucleus, leading to certain changes in the growth of the cell. If the nucleus be the bearer of the past history of the individual, the scheme here sketched would accord with the adaptive character of normal reactions, and would fall into line with what we know of the regulation of actions in the higher organisms. Pfeffer ("Physiology of Plants," Eng. trans., iii., 10) has briefly discussed the possibility of thus considering the nucleus as a reflex centre, and has pointed out difficulties in the way of accepting such a view as universally holding good. Delage ("L'Hérédite," 2nd Edit., 1903, p. 88) gives a good summary of the evidence which induces him to deny the mastery of the cell by the nucleus. Driesch, however ("Analytische Theorie der organischen Entwicklung," 1894, p. 81), gives reasons for believing that the cytoplasm is the receptive region, while the nucleus is responsible for the reaction, and it is on this that he bases his earlier theory of ontogeny.

* "Die Mneme, als erhaltendes Prinzip im Wechsel des organischen Geschehens," von Richard Semon, 1te Auflage, Leipzig, 1904, 2te Auflage, 1908. It is a pleasure to express my indebtedness to this work, as well as for the suggestions and criticisms which I owe to Professor Semon personally.

a habit built by the alternation of day and night. The plant normally drops its leaves at the stimulus of darkness and raises them at the stimulus of light. But here we see the leaves rising and falling in the absence of the accustomed stimulation. Since this change of position is not due to external conditions it must be the result of the internal conditions which habitually accompany the movement. This is the characteristic *par excellence* of habit—namely, a capacity, acquired by repetition, of reacting to a fraction of the original environment. We may express it in simpler language. When a series of actions are compelled to follow each other by applying a series of stimuli they become organically tied together, or *associated*, and follow each other automatically, even when the whole series of stimuli are not acting. Thus in the formation of habit *post hoc* comes to be equivalent to *propter hoc*. Action B automatically follows action A, because it has repeatedly been compelled to follow it.

This may be compared with Herbert Spencer's description of an imaginary case ("Psychology," 2nd edit., 1870, vol. i., p. 435), that of a simple aquatic animal which contracts its tentacles on their being touched by a fish or a bit of seaweed washed against it. If such a creature is also sensitive to light the circumstances under which contraction takes place will be made up of two stimuli—those of light and of contact—following each other in rapid succession. And, according to the above statement of the essential character of associative habit, it will result that the light-stimulus alone may suffice, and the animal will contract without being touched.

Jennings ("Behaviour of the Lower Organisms," 1906, p. 289) has shown that the basis of memory by association exists in so low an organism as the infusorian *Stentor*. When the animal is stimulated by a jet of water containing carmine in suspension, a physiological state A is produced, which, however, does not immediately lead to a visible reaction. As the carmine stimulus is continued or repeated, state B is produced, to which the *Stentor* reacts by bending to one side. After several repetitions of the stimulus, state C is produced, to which the animal responds by reversing its ciliary movement, and C finally passes into D, which results in the *Stentor* contracting into its tube. The important thing is that after many repetitions of the above treatment the organism "contracts at once as soon as the carmine comes in contact with it." In other words, states B and C are apparently omitted, and A passes directly into D, *i.e.*, into the state which gives contraction as a reaction. Thus we have in an infusorian a case of short-circuiting precisely like the case which has been quoted from Herbert Spencer as illustrating association. But Jennings' case has the advantage of being based on actual observation. He generalises the result as the "law of the resolution of physiological states" in the following words:—"The resolution of one physiological state into another becomes easier and more rapid after it has taken place a number of times." He goes on to point out that the operation of this law is seen in the higher organisms, "in the phenomena which we commonly call memory, association, habit-formation, and learning."

In spite of this evidence of mnemic power in the simplest of organisms, objections will no doubt be made to the statement that association of engrams can occur in plants.

Pfeffer, whose authority none can question, accounts for the behaviour of sleeping-plants principally on the more general ground that when any movement occurs in a plant there is a tendency for it to be followed by a reversal—a swing of the physiological pendulum in the other direction. Pfeffer* compares it to a released spring which makes several alternate movements before it settles down to equilibrium. But the fact that the return movements occur at the same time-intervals as the stimuli is obviously the striking feature of the case. If the pendulum-like swing always tended to occur naturally in a twelve hours' rhythm it would be a different matter. But Pfeffer has shown that a rhythm of six hours can equally well be built up. And the experiments of Miss Pertz and myself ("Annals of Botany," 1892 and 1903) show that a half-hourly or quarter-hourly rhythm can be produced by alternate geotropic stimulation.

We are indebted to Keeble (Gamble and Keeble, *Q. J. Mic. Science*, xlvii., p. 401) for an interesting case of apparent habit among the lower animals. *Convoluta roscoffensis*, a minute wormlike creature found on the coast of Brittany, leads a life dependent on the ebb and flow of the sea. When the tide is out the *Convoluta* come to the surface, showing themselves in large green patches. As the rising tide begins to cover them they sink down into safer quarters. The remarkable fact is that when kept in an aquarium, and therefore removed from tidal action, they continue for a short time to perform rhythmic movements in time with the tide.

Let us take a human habit, for instance that of a man who goes a walk every day and turns back at a given mile-post. This becomes habitual, so that he reverses his walk automatically when the limit is reached. It is no explanation of the fact that the stimulus which makes him start from home includes his return—that he has a mental return-ticket. Such explanation does not account for the point at which he turns, which as a matter of fact is the result of association. In the same way a man who goes to sleep will ultimately wake; but the fact that he wakes at four in the morning depends on a habit built up by his being compelled to rise daily at that time. Even those who will deny that anything like association can occur in plants cannot deny that in the continuance of the nyctitropic rhythm in constant conditions we have, in plants, something which has general character of habit, *i.e.*, a rhythmic action depending on a rhythmic stimulus that has ceased to exist.

On the other hand, many will object that even the simplest form of association implies a nervous system. With regard to this objection it must be remembered that plants have two at least of the qualities characteristic of animals—namely, extreme sensitiveness to certain agencies and the power of transmitting stimuli from one part to another of the plant body. It is true that there is no central nervous system, nothing but a complex system of nuclei; but these have some of the qualities of nerve cells, while intercommunicating protoplasmic threads may play the part of nerves. Spencer ("Psychology," 2nd edit., vol. i., p. 615) bases the power of association on the fact that every discharge conveyed by a nerve "leaves it in a state for conveying a subsequent like discharge with less resistance." Is it not possible that the same thing may be as true of plants as it apparently is of infusoria? We have seen reasons to suppose that the "internal conditions" or "physiological states" in plants are of the nature of engrams, or residual effects of external stimuli, and such engrams may become associated in the same way.

There is likely to be another objection to my assumption that a simple form of associated action occurs in plants—namely, that association implies consciousness. It is impossible to know whether or not plants are conscious; but it is consistent with the doctrine of continuity that in all living things there is something psychic, and if we accept this point of view we must believe that in plants there exists a faint copy of what we know as consciousness in ourselves (see James Ward, "Naturalism and Agnosticism," vol. i., Lecture X.).

I am told by psychologists that I must define my point of view. I am accused of occupying that unscientific position known as "sitting on the fence." It is said that, like other biologists, I try to pick out what suits my purpose from two opposite schools of thought—the psychological and the physiological.

What I claim is that, as regards reaction to environment, a plant and a man must be placed in the same great class, in spite of the obvious fact that as regards complexity of behaviour the difference between them is enormous. I am

* See Pfeffer, *Abhandl. K. Sachs. Ges.*, Bd. xxx., 1907. It is impossible to do justice to Pfeffer's point of view in the above brief statement.

not a psychologist, and I am not bound to give an opinion as to how far the occurrence of definite actions in response to stimulus is a physiological and how far a psychological problem. I am told that I have no right to assume the neural series of changes to be the cause of the psychological series, though I am allowed to say that neural changes are the universal concomitants of psychological change. This seems to me, in my ignorance, an unsatisfactory position. I find myself obliged to believe that the mnemic quality in all living things (which is proved to exist by direct experiment) must depend on the physical changes in protoplasm, and that it is therefore permissible to use these changes as a notation in which the phenomena of habit may be expressed.

Habit illustrated by Morphology.

We have hitherto been considering the mnemic quality of movements; but, as I have attempted to show, morphological changes are reactions to stimulation of the same kind as these temporary changes. It is indeed from the morphological reactions of living things that the most striking cases of habit are, in my opinion, to be found.

The development of the individual from the germ-cell takes place by a series of stages of cell-division and growth, each stage apparently serving as a stimulus to the next, each unit following its predecessor like the movements linked together in an habitual action performed by an animal.

My view is that the rhythm of ontogeny is actually and literally a habit. It undoubtedly has the feature which I have described as pre-eminently characteristic of habit, viz., an automatic quality which is seen in the performance of a series of actions in the absence of the complete series of stimuli to which they (the stages of ontogeny) were originally due. This is the chief point on which I wish to insist—I mean that the resemblance between ontogeny and habit is not merely superficial, but deeply seated. It was with this conclusion in view that I dwelt, at the risk of being tedious, on the fact that memory has its place in the morphological as well as in the temporary reactions of living things. It cannot be denied that the ontogenetic rhythm has the two qualities observable in habit—namely, a certain degree of fixity or automaticity, and also a certain variability. A habit is not irrevocably fixed, but may be altered in various ways. Parts of it may be forgotten or new links may be added to it. In ontogeny the fixity is especially observable in the earlier, the variability in the later, stages. Mr. Darwin has pointed out that "on the view that species are only strongly marked and fixed varieties, we might expect often to find them still continuing to vary in those parts of their structure which have varied within a moderately recent period." These remarks are in explanation of the "notorious" fact that specific are more variable than generic character—a fact for which it is "almost superfluous to adduce evidence" ("Origin of Species," 6th edit., p. 122). This, again, is what we find in habit: take the case of a man who, from his youth up, has daily repeated a certain form of words. If in middle life an addition is made to the formula, he will find the recently acquired part more liable to vary than the rest.

Again, there is the wonderful fact that, as the ovum develops into the perfect organism, it passes through a series of changes which are believed to represent the successive forms through which its ancestors passed in the process of evolution. This is precisely paralleled by our own experience of memory, for it often happens that we cannot reproduce the last learned verse of a poem without repeating the earlier part; each verse is suggested by the previous one and acts as a stimulus for the next. The blurred and imperfect character of the ontogenetic version of the phylogenetic series may at least remind us of the tendency to abbreviate by omission what we have learned by heart.

In all bi-sexual organisms the ontogenetic rhythm of the offspring is a combination of the rhythms of its parents. This may or may not be visible in the offspring; thus in the crossing of two varieties the mongrel assumes the character of the prepotent parent. Or the offspring may show a blend of both parental characters. Semon "Die Mneme," 2nd Ed., pp. 147, 221, 303, 345) uses as a model the two versions of Goethe's poem:—

"Ueber allen Gipfeln, ist Ruh, in allen { Wäldern, hörest du, keinen Hauch. / Wipfeln, spürest du, kaum einen Hauch."

One of these terminations will generally be prepotent, probably the one that was heard first or heard most often. But the cause of such prepotency may be as obscure as the corresponding occurrence in the formation of mongrels. We can only say that in some persons the word "allen" releases the word "Wäldern," while in others, it leads up to "Wipfeln." Again, a mixture of the terminations may occur leading to such a mongrel form as: "in allen Wäldern hörest du kaum einen Hauch." The same thing is true of music; a man with an imperfect memory easily interpolates in a melody a bar that belongs elsewhere. In the case of memory the introduction of a link from one mental rhythm into another can only occur when the two series are closely similar, and this may remind us of the difficulty of making a cross between distinctly related forms.

Enough has been said to show that there is a resemblance between the two rhythms of development and of memory; and that there is at least a *prima facie* case for believing them to be essentially similar. It will be seen that my view is the same as that of Hering, which is generally described as the identification of memory and inheritance.* Hering says that "between the *me* of to-day and the *me* of yesterday lie night and sleep, abysses of unconsciousness; nor is there any bridge but memory with which to span them." And in the same way he claims that the abyss between two generations is bridged by the unconscious memory that resides in the germ cells. It is also the same as that of Semon, and to a great extent as that of Rignano ("Sur la Transmissibilité des Caractères Acquis," Paris, 1906). I, however, prefer at the moment to limit myself to asserting the identity of ontogeny and habit, or, more generally, to the assertion in Semon's phraseology, that ontogeny is a mnemic phenomenon.

Evolution, in its modern sense, depends on a change in the ontogenetic rhythm. This is obvious, since if this rhythm is absolutely fixed, a species can never give rise to varieties. This being so, we have to ask *in what ways* the ontogenetic rhythm can be altered. An habitual action, for instance, a trick learned by a dog, may be altered by adding new accomplishments; at first the animal will persist in finishing his performance at the old place, but at last the extended trick will be bonded into a rhythm of actions as fixed as was the original simpler performance. May we not believe that this is what has occurred in evolution.

We know from experiment that a plant may be altered in form by causes acting on it during the progress of development. Thus a beech tree may be made to develop different forms of leaves by exposing it to sunshine or to shade. The ontogeny is different in the two cases, and what is of special interest is, that there exist shade-loving plants in which a structure similar to that of the shaded beech-leaf is apparently typical of the species, but on this point it is necessary to speak with caution. In the same way Goebel points out that in some orchids the assimilating roots take on a flattened form when exposed to sunlight, but in others this morphological change has become automatic, and occurs even in darkness (Goebel's "Organography of Plants," Part II., p. 285).

* Everyone who deals with this subject must take his stand on the foundation laid by Hering in his celebrated Address given at Vienna in 1870, and reprinted in No. 148 of Ostwald's "Exakt Klassiker." The passage quoted (p. 14) is from Samuel Butler's translation of Hering in "Unconscious Memory," 1880, p. 110. Butler had previously elaborated the view that "we are one person with our ancestors" in his entertaining book "Life and Habit," 1878, and this was written in ignorance of Hering's views.

Such cases suggest at least the possibility of varieties arising as changes in or additions to the later stages of ontogeny. This is, briefly given, the epigenetic point of view.

But there is another way of looking at the matter—namely, that upheld by Galton and Weismann. According to this view ontogeny can only be changed by a fundamental upset of the whole system—namely, by an alteration occurring in its first stage, the germ cell, and this view is now very generally accepted.

The same type of change may conceivably occur in memory or habit; that is, the rhythm as a whole may be altered by some cause acting on the nerve-centres connected with the earlier links of the series. The analogy is not exact, but such an imaginary case is at least of a different type from a change in habit consisting in the addition of a new link or the alteration of one of the latest formed links. If we were as ignorant of the growth of human actions as we are of variation, we might have a school of naturalists asserting that all changes in habit originate in the earliest link of the series. But we know that this is not the case. On the other hand, I fully admit that the structure of an ovum may in this way be altered, and give rise to a variation which may be the starting-point of a new species.

But how can a new species originate according to an epigenetic theory? How can a change in the latter stages of ontogeny produce a permanent alteration in the germ-cells? Our answer to this question will depend on our views of the structure of the germ-cells. According to the mnemic theory they have the quality which is found in the highest perfection in nerve-cells, but is at the same time a character of all living matter—namely, the power of retaining the residual effects of former stimuli and of giving forth or reproducing under certain conditions an echo of the original stimulus. In Semon's phraseology germ-cells must, like nerve-cells, contain engrams, and these engrams must be (like nerve-engrams) bonded together by association, so that they come into action one after another in a certain order automatically, *i.e.*, in the absence of the original stimuli.

This seems to me the strength of the mnemic theory—namely, that it accounts for the preformed character of germ-cells by the building up in them of an organised series of engrams. But if this view has its strength, it has also its weakness. Routine can only be built up by repetition, but each stage in ontogeny occurs only once in a lifetime. Therefore if ontogeny is a routine each generation must be mnemically connected with the next. This can only be possible if the germ-cells are, as it were, in telegraphic communication with the whole body of the organism; so that as ontogeny is changed by the addition of new characters, new engrams are added to the germ-cell.

Thus, in fact, the mnemic theory of development depends on the possibility of what is known as somatic inheritance or the inheritance of acquired characters. This is obvious to all those familiar with the subject, but to others it may not be so clear. Somatic inheritance is popularly interesting in relation to the possible inherited effects of education, or of mutilations, or of the effects of use and disuse. It is forgotten that it may be, as I have tried to show, an integral part of all evolutionary development.

Weismann's Theory.

Everyone must allow that if Weismann's theory of inheritance is accepted we cannot admit the possibility of somatic inheritance. This may be made clear to those unfamiliar with the subject by an illustration taken from the economy of an ant's nest or beehive. The queen* on whom depends the future of the race is cut off from all active experience of life: she is a mere reproducing machine, housed, fed, and protected by the workers. But these, on whom falls the burden of the struggle for life and the experience of the world generally, are sterile, and take no direct share in the reproduction of the species. The queen represents Weismann's germ-plasm, the workers are the body or soma. Now imagine the colony exposed to some injurious change in environment; the salvation of the species will depend on whether or no an improved pattern of worker can be produced. This depends on the occurrence of appropriate variations, so that the queen bee and the drones, on whom this depends, are of central importance. On the other hand, any change occurring in the workers, for instance, increased skill due to practice in doing their work or changes in their structure due to external conditions, cannot possibly be inherited, since workers are absolutely cut off from the reproduction of the race. According to Weismann, there is precisely the same bar to the inheritance of somatic change.

The racial or phyletic life of all organisms is conceived by him as a series of germ-cells whose activity is limited to varying, and whose survival in any generation depends on the production of a successful soma or body capable of housing, protecting, and feeding the germ-cell. Most people would *à priori* declare that a community where experience and action are separated must fail. But the bee's nest, which must be allowed to be something more than an illustration of Weismann's theory, proves the contrary.

It is clear that there must be war to the knife between the theory of Weismann and that of the somatists—to coin a name for those who believe in the inheritance of acquired characters. A few illustrations may be given of the strength of Weismann's position. Some trick or trivial habit appears in two successive generations, and the son is said to inherit it from his father. But this is not necessarily a case of somatic inheritance, since, according to Weismann, the germ-plasm of both father and son contained the potentiality of the habit in question. If we keep constantly in view Weismann's theory of continuity, the facts which are supposed to prove somatic inheritance cease to be decisive.

Weismann has also shown by means of his hypothesis of "simultaneous stimulation"* the unconvincingness of a certain type of experiment. Thus Fischer showed that when chrysalids of *Arctia caja* are subjected to low temperature a certain number of them produce dark coloured insects; and further that these moths mated together yield dark coloured offspring. This has been held to prove somatic inheritance, but Weismann points out that it is explicable by the low temperature having an identical effect on the colour-determinants existing in the wing-rudiments of the pupa, and on the same determinants occurring in the germ-cells.

It does not seem to me worth while to go in detail into the evidence by which somatists strive to prove their point, because I do not know of any facts which are really decisive. That is to say, that though they are explicable as due to somatic inheritance, they never seem to me absolutely inexplicable on Weismann's hypothesis. But, as already pointed out, it is not necessary to look for special facts and experiments, since if the mnemic theory of ontogeny is accepted the development of every organism in the world depends on somatic inheritance.

I fully acknowledge the strength of Weismann's position; I acknowledge also most fully that it requires a stronger man than myself to meet that trained and well-tried fighter. Nevertheless, I shall venture on a few remarks. It must be rembered that, as Romanes pointed out ("An Examination of Weismann," 1893, pp. 169, 170), Weismann has greatly strengthened his theory of heredity by giving up the absolute stability and perpetual continuity of germ-plasm. Germ-plasm is no longer that mysterious entity, immortal and self-contained, which used to suggest a physical soul. It is no longer the aristocrat it was when its only activity was dependent on its protozoan ancestors,

* Nor do the drones share the activity of the workers.

* I borrow this convenient expression from Plate's excellent book, "Ueber die Bedeutung des Darwin'schen Selectionsprincips," 1903, p. 81.

when it reigned absolutely aloof from its contemporary subjects. The germ-plasm theory of to-day is liberalised, though it is not so democratic as its brother sovereign Pangenesis, who reigns, or used to reign, by an elaborate system of proportional representation. But in spite of the skill and energy devoted to its improvement by its distinguished author, Weismannism fails, in my opinion, to be a satisfactory theory of evolution.

All such theories must account for two things which are parts of a single process but may logically be considered separately:—(i.) The fact of ontogeny, namely, that the ovum has the capacity of developing into a certain more or less predetermined form; (ii.) the fact of heredity—the circumstance that this form is approximately the same as that of the parent.

The doctrine of pangenesis accounts for heredity, since the germ-cells are imagined as made up of gemmules representing all parts of the adult; but it does not account for ontogeny, because there seems to me no sufficient reason why the gemmules should become active in a predetermined order unless, indeed, we allow that they do so by habit, and then the doctrine of pangenesis become a variant of the mnemic theory.

The strength of Weismann's theory lies in its explanation of heredity. According to the doctrine of continuity, a fragment of the germ-plasm is, as it were, put on one side and saved up to make the germ-cell of the new generation, so that the germ-cells of two successive generations are made of the same material. This, again, depends on Weismann's belief that when the ovum divides, the two daughter cells are not identical; that, in fact, the fundamental difference between soma and germ-cells begins at this point. But this is precisely where many naturalists whose observations are worthy of all respect differ from him. Weismann's theory is therefore threatened at the very foundation.

Even if we allow Weismann's method of providing for the identity between the germ-cell of two successive generations, there remains, as above indicated, a greater problem namely, that of ontogeny. We no longer look at the potentiality of a germ-cell as Caliban looked on Setebos, as something essentially incomprehensible ruling the future in an unknown way—"just choosing so." If the modern germ-cell is to have a poetic analogue it must be compared to a Pandora's box of architectonic sprites which are let loose in definite order, each serving as a master-builder for a prescribed stage of ontogeny. Weismann's view of the mechanism by which his determinants—the architectonic sprites—come into action in due order is, I assume, satisfactory to many, but I confess that I find it difficult to grasp. The orderly distribution of determinants depends primarily on their arrangement in the ids, where they are held together by "vital affinities." They are guided to the cells on which they are to act by differential divisions, in each of which the determinants are sorted into two unequal lots. They then become active, *i.e.*, break up into biophores, partly under the influence of liberating stimuli and partly by an automatic process. Finally, the biophores communicate a "definite vital force" to the appropriate cells ("The Evolution of Theory," Eng. trans., i., 373 *et seq.*). This *may* be a description of what happens; but inasmuch as it fails to connect the process of ontogeny with physiological processes of which we have definite knowledge, it does not to me seem a convincing explanation.

For myself I can only say that I am not satisfied with Weismann's theory of heredity or of ontogeny. As regards the first, I incline to deny the distinction between germ and soma, to insist on the plain facts that the soma is continuous with the germ-cell, and that the somatic cells may have the same reproductive qualities as the germ-cells (as is proved by the facts of regeneration); that, in fact, the germ-cell is merely a specialised somatic cell and has the essential qualities of the soma. With regard to ontogeny, I have already pointed out that Weismann does not seem to explain its automatic character.

The Mnemic Theory.

If the mnemic theory is compared with Weismann's views it is clear that it is strong precisely where these are weakest—namely, in giving a coherent theory of the rhythm of development. It also bears comparison with all theories in which the conception of determinants occurs. Why should we make elaborate theories of hypothetical determinants to account for the potentialities lying hidden in the germ-cell, and neglect the only determinants of whose existence we have positive knowledge (though we do not know their precise nature)? We know positively that by making a dog sit up and then giving him a biscuit we build up something in his brain in consequence of which a biscuit becomes the stimulus to the act of sitting. The mnemic theory assumes that the determinants of morphological change are of the same type as the structural alteration wrought in the dog's brain.

The mnemic theory—at any rate that form of it held by Semon and by myself—agrees with the current view, viz., that the nucleus is the centre of development, or, in Semon's phraseology, that the nucleus contains the engrams in which lies the secret of the ontogenetic rhythm. But the mode of action of the mnemic nucleus is completely different from that of Weismann. He assumes that the nucleus is disintegrated in the course of development by the dropping from it of the determinants which regulate the manner of growth of successive groups of cells. But if the potentiality of the germ nucleus depends on the presence of engrams, if, in fact, its function is comparable to that of a nerve-centre, its capacity is not diminished by action; it does not cast out engrams from its substance as Weismann's nucleus is assumed to drop armies of determinants. The engrams are but cut deeper into the records, and more closely bonded one with the next. The nucleus, considered as a machine, does not lose its component parts in the course of use. We shall see, later on, that the nuclei of the whole body may, on the mnemic theory, be believed to become alike. The fact that the mnemic theory allows the nucleus to retain its repeating or reproductive or mnemic quality supplies the element of continuity. The germ-cell divides and its daughter cells form the tissues of the embryo, and in this process the original nucleus has given rise to a group of nuclei; these, however, have not lost their engrams, but retain the potentiality of the parent nucleus. We need not therefore postulate the special form of continuity which is characteristic of Weismann's theory.

We may say therefore that the mnemic hypothesis harmonises with the facts of heredity and ontogeny. But the real difficulties remain to be considered, and these, I confess, are of a terrifying magnitude.

The first difficulty is the question how the changes arising in the soma are, so to speak, telegraphed to the germ-cells. Hering allows that such communication must at first seem highly mysterious.* He then proceeds to show how by the essential unity and yet extreme ramification of the nervous system "all parts of the body are so connected that what happens in one echoes through the rest, so that from the disturbance occurring in any part some notification, faint though it may be, is conveyed to the most distant parts of the body."

A similar explanation is given by Nägeli. He supposes that adaptive, in contradistinction to organic, characters are produced by external causes; and since these characters are hereditary there must be communication between the seat of adaptation and the germ-cells. This telegraphic effect is supposed to be effected by the network of idioplasm which traverses the body, in the case of plants by the intercellular protoplasmic threads.

Semon faces the difficulty boldly. When a new character appears in the body of an organism, in response to changing environment, Semon assumes that a new

* E. Hering in Ostwald's "Klassiker der exakten Wissenschaften," No. 148, p. 14; see also S. Butler's translation in "Unconscious Memory," p. 119.

engram is added to the nuclei in the part affected; and that, further, the disturbance tends to spread to all the nuclei of the body (including those of the germ-cells), and to produce in them the same change. In plants the flow must be conceived as travelling by intercellular plasmic threads, but in animals primarily by nerve-trunks. Thus the reproductive elements must be considered as having in some degree the character of nerve-cells. So that, for instance, if we are to believe that an individual habit may be inherited and appear as an instinct, the repetition of the habit will not merely mean changes in the central nervous system, but also corresponding changes in the germ-cells. These will be, according to Semon, excessively faint in comparison to the nerve-engrams, and can only be made efficient by prolonged action. Semon lays great stress on the slowness of the process of building up efficient engrams in the germ-cells.

Weismann* speaks of the impossibility of germinal engrams being formed in this way. He objects that nerve-currents can only differ from each other in intensity, and therefore there can be no communication of potentialities to the germ-cell. He holds it to be impossible that somatic changes should be telegraphed to the germ-cell and be reproduced ontogenetically—a process which he compares to a telegram despatched in German and arriving in Chinese. According to Semon,† what radiates from the point of stimulation in the soma is the primary excitation set up in the somatic cells; if this is so, the radiating influence will produce the same effect on all the nuclei of the organism. My own point of view is the following:—In a plant (as already pointed out) the ectoplasm may be compared to the sense-organ of the cell, and the primary excitation of the cell will be a change in the ectoplasm; but since cells are connected by ectoplasmic threads the primary excitation will spread and produce in other cells a faint copy of the engram impressed on the somatic cells originally stimulated. But in all these assumptions we are met by the question to which Weismann has called attention—namely, whether nervous impulses can differ from one another in *quality*?‡ The general opinion of physiologists is undoubtedly to the opposite effect—namely, that all nervous impulses are identical in quality. But there are notable exceptions; for instance, Hering ("Zur Theorie der Nerventhätigkeit, Akademische Vortrag," 1898, Veit, Leipzig), who strongly supports what may be called the qualitative theory. I am not competent to form an opinion on the subject, but I confess to being impressed by Hering's argument that the nerve cell and nerve fibre, as parts of one individual (the neuron), must have a common irritability. On the other hand, there is striking evidence, in Langley's experiments on the cross-grafting of efferent nerves, that here at least nerve impulses are interchangeable and therefore identical in quality (*Proc. Roy. Soc.*, 1904, p. 99; *Journal of Physiology*, xxiii., p. 240, and xxxi., p. 365). The state of knowledge as regards afferent nerves is, however, more favourable to my point of view. For the difficulties that meet the physiologist—especially as regards the nerves of smell and hearing—are so great that it has been found simpler to assume differences in impulse-quality rather than attempt an explanation of the facts on the other hypothesis (see Nagel, "Handbuch der Physiologie des Menschen," 1905, iii., pp. 1—15).

On the whole it may be said that, although the trend of physiological opinion is against the general existence of qualitative difference in nerve-impulses, yet the question cannot be said to be settled either one way or the other.

Another obvious difficulty is to imagine how within a single cell the engrams or potentialities of a number of actions can be locked up. We can only answer that the nucleus is admittedly very complex in structure. It may be added (but this not an answer) that in this respect it claims no more than its neighbours; it need not be more complex than Weismann's germ-plasm. One conceivable simplification seems to be in the direction of the pangenes of De Vries. He imagines that these heritage-units are relatively small in number, and that they produce complex results by combination, not by each being responsible for a minute fraction of the total result (De Vries, "Intracellular Pangenesis," p. 7). They may be compared to the letters of the alphabet which by combination make an infinity of words.* Nägeli (Nägeli's "Abstammungslehre, 1884, p. 73) held a similar view. "To understand heredity," he wrote, "we do not need a special independent symbol for every difference conditioned by space, time, or quality, but a substance which can represent every possible combination of differences by the fitting together of a limited number of elements, and which can be transformed by permutations into other combinations." He applied (*loc. cit.*, p. 59) the idea of a combination of symbols to the telegraphic quality of his idioplasm. He suggests that as the nerves convey the most varied perceptions of external objects to the central nervous system, and there create a coherent picture, so it is not impossible that the idioplasm may convey a combination of its local alterations to other parts of the organism.

Another theory of simplified telegraphy between soma and germ-cell is given by Rignano.† I regret that the space at my command does not permit me to give a full account of his interesting speculation on somatic inheritance. It resembles the theories of Hering, Butler, and Semon in postulating a quality of living things, which is the basis both of memory and inheritance. But it differs from them in seeking for a physical explanation or model of what is common to the two. He compares the nucleus to an electric accumulator which in its discharge gives out the same sort of energy that it has received. How far this is an allowable parallel I am not prepared to say, and in what follows I have given Rignano's results in biological terms. What interests me is the conclusion that the impulse conveyed to the nucleus of the germ-cell is, as far as results are concerned, the external stimulus. Thus, if a somatic cell (A) is induced by an external stimulus (S) acting on the nucleus to assume a new manner of development, a disturbance spreads through the organism, so that finally the nuclei of the germ-cells are altered in a similar manner. When the cellular descendants of the germ-cells reach the same stage of ontogeny as that in which the original stimulation occurred, a stimulus comes into action equivalent to S as regards the results it is capable of producing. So that the change originally wrought in cell A by the actual stimulus S is now reproduced by what may be called an inherited stimulus. But when A was originally affected other cells, B, C, D, may have reacted to S by various forms of growth. And therefore when during the development of the altered germ-cell something equivalent to S comes into play, there will be induced, not merely the original change in the development of A, but also the changes which were originally induced in the growth of B, C, D. Thus, according to Rignano, the germ-nucleus releases a number of developmental processes, each of which would, according to Weismann, require a separate determinant.

If the view here given is accepted, we must take a new view of Weismann's cases of *simultaneous stimulation*, *i.e.*, cases like Fischer's experiments on *Arctia caja*, which he does not allow to be somatic inheritance. If we are right in saying that, the original excitation of the soma is transferred to the germ-cell, and it does not matter whether the stimulus is transferred by "telegraphy," or whether a given cause, *e.g.*, a low temperature, acts simultaneously

* Weismann, "The Evolution Theory," 1904, vol. ii., p. 63; also his "Richard Semon's 'Mneme' und die Vererbung erworbener Eigenschaften," in the *Archiv für Rassen- und Gesellschafts-Biologie*, 1906. Semon has replied in the same journal for 1907.

† Semon ("Mneme," ed. i, p. 142), does not, however, consider it proved that the nucleus is necessarily the smallest element in which the whole inheritance resides. He refers especially to the regeneration of sections of *Stentor* which contain mere fragments of the nucleus.

‡ I use this word in the ordinary sense without reference to what is known as *modality*.

* I take this comparison from Lotsy's account of De Vries' theory. Lotsy, "Vorlesungen über Deszendenztheories," 1906, i., p. 98.

† For what is here given I am partly indebted to Signor Rignano's letters.

on soma and germ-cell. In both cases we have a given alteration produced in the nuclei of the soma and the germ-cell. Nägeli used the word *telegraphy* to mean a dynamic form of transference, but he did not exclude the possibility of the same effect being produced by the movement of chemical substances, and went so far as to suggest that the sieve tubes might convey such stimuli in plants. In any case this point of view deserves careful consideration (see Semon, *Archiv. f. Rassen- und Gesellschafts-Biologie*, 1907, p. 39).

Still another code of communication seems to me to be at least conceivable. One of the most obvious characteristics of animal life is the guidance of the organism by certain groups of stimuli, producing either a movement of seeking—positive reaction (see Jennings, "Behaviour of the Lower Organisms")—or one of avoidance—negative reaction. Taking the latter as being the simplest, we find that in the lowest as in the highest organisms a given reaction follows each one of a number of diverse conditions which have nothing in common save that they are broadly harmful in character. We withdraw our hands from a heated body, a prick, a corrosive substance, or an electric shock. The interesting point is that it is left to the organism to discover by the method of trial and error the best means of dealing with a subinjurious stimulus. May we not therefore say that the existence of pleasure and pain simplifies inheritance? It certainly renders unnecessary a great deal of detailed inheritance. The innumerable appropriate movements performed by animals are broadly the same as those of their parents, but they are not necessarily inherited in every detail; they are rather the unavoidable outcome of hereditary but unspecialised sensitiveness. It is as though heredity were arranged on a code-system instead of by separate signals for every movement of the organism.

It may be said that in individual life the penalty of failure is pain, but that the penalty for failure in ontogenetic morphology is death. But it is only because pain is the shadow cast by Death as he approaches that it is of value to the organism. Death would be still the penalty of creatures that had not acquired this sensitiveness to the edge of danger. Is it not possible that the sensitiveness to external agencies by which structural ontogeny is undoubtedly guided may have a similar quality, and that morphological variations may also be reactions to the edge of danger. But this is a point of view I cannot now enter upon.

It may be objected that the inheritance of anything so complex as an instinct is difficult to conceive on the mnemic theory. Yet it is impossible to avoid suspecting that at least some instincts originate in individual acquirements, since they are continuous with habits gained in the lifetime of the organism. Thus the tendency to peck at any small object is undoubtedly inherited; the power of distinguishing suitable from unsuitable objects is gained by experience. It may be said that the engrams concerned in the pecking instinct cannot conceivably be transferred from the central nervous system to the nucleus of the germ-cells. To this I might answer that this is not more inconceivable than Weismann's assumption that the germ-cell chances to be so altered that the young chicken pecks instinctively. Let us consider another case of what appears to be an hereditary movement. Take, for instance, the case of a young dog, who in fighting bites his own lips. The pain thus produced will induce him to tuck up his lips out of harm's way. This protective movement will become firmly associated with, not only the act of fighting but with the remembrance of it, and will show itself in the familiar snarl of the angry dog. This movement is now, I presume, hereditary in dogs, and is so strongly inherited by ourselves (from simiam ancestors) that a lifting of the corner of the upper lip is a recognised signal of adverse feeling. Is it really conceivable that the original snarl is due to that unspecialised stimulus we call pain, whereas the inherited snarl is due to fortuitous upsets of the determinants in the germ cell?

I am well aware that many other objections may be advanced against the views I advocate. To take a single instance, there are many cases where we should expect somatic inheritance, but where we look in vain for it. This difficulty, and others equally important, must for the present be passed over. Nor shall I say anything more as to the possible means of communication between the soma and the germ-cells. To me it seems conceivable that some such telegraphy is possible. But I shall hardly wonder if a majority of my hearers decide that the available evidence in its favour is both weak and fantastic. Nor can I wonder that, apart from the problem of mechanism, the existence of somatic inheritance is denied for want of evidence. But I must once more insist that according to the mnemic hypothesis, somatic inheritance lies at the root of all evolution. Life is a gigantic experiment which the opposing schools interpret in opposite ways. I hope that in this dispute both sides will seek out and welcome decisive results. My own conviction in favour of somatic inheritance rests primarily on the automatic element in ontogeny. It seems to me certain that in development we have an actual instance of habit. If this is so, somatic inheritance must be a *vera causa*. Nor does it seem impossible that memory should rule the plasmic link which connects successive generations—the true miracle of the camel passing through the eye of a needle—since, as I have tried to show, the reactions of living things to their surroundings exhibit in the plainest way the universal presence of a mnemic factor.

We may fix our eyes on phylogeny and regard the living world as a great chain of forms, each of which has learned something of which its predecessors were ignorant; or we may attend rather to ontogeny, where the lessons learned become in part automatic. But we must remember that the distinction between phylogeny and ontogeny is an artificial one, and that routine and aquisition are blended in life.*

The great engine of natural selection is taunted nowadays, as it was fifty years ago, with being merely a negative power. I venture to think that the mnemic hypothesis of evolution makes the positive value of natural selection more obvious. If evolution is a process of drilling organisms into habits, the elimination of those that cannot learn is an integral part of the process, and is no less real because it is carried out by a self-acting system. It is surely a positive gain to the harmony of the universe that the discordant strings should break. But natural selection does more than this; and just as a trainer insists on his performing dogs accommodating themselves to conditions of increasing complexity, so does natural selection pass on its pupils from one set of conditions to other and more elaborate tests, insisting that they shall endlessly repeat what they have learned and forcing them to learn something new. Natural selection attains in a blind, mechanical way the ends gained by a human breeder; and by an extension of the same metaphor it may be said to have the power of a trainer—of an automatic master with endless patience and all time at his disposal.

New Crystallised Sugar, Perseulose, with Seven Carbon Atoms.—Gabriel Bertrand.—The oxidation of perseite by means of the bacteria of sorbose yields a new crystalline sugar to which the author has given the name perseulose. It resembles glucose in appearance, and its formula is $C_7H_{14}O_7$. It is lævorotatory, and, like other reducing sugars, possesses the property of multirotation. Its reducing power is inferior to that of glucose but greater than that of sorbose. It yields a crystalline osazone, $C_{19}H_{24}N_4O_5$. Perseulose is the first reducing sugar with seven atoms of carbon produced by a living cell, which has been isolated.—*Comptes Rendus*, cxlvii., No. 3.

* This subject is dealt with in a very interesting manner in Professor James Ward's forthcoming lectures on the "Realm of Ends." Also in his article on "Mechanism and Morals," in the *Hibbert Journal*, October, 1905, p. 92; and in his article on Psychology in the "Encyclopædia Britannica," 1886, xx., p. 44.

THE ELECTROLYTIC PROPERTIES OF DILUTE SOLUTIONS OF SULPHURIC ACID.*

By W. C. D. WHETHAM, M.A. F.R.S., and H. H. PAINE, B.A., Trinity College, Cambridge.

(Continued from p. 100).

Part II.—*By* H. H. Paine.

1. *Method of Experiment.*

The migration constant was determined for solutions of sulphuric acid of concentration varying from 0·1 to 0·00005 normal. A current, the strength of which was determined by means of a standardised galvanometer, was passed through the migration cell, and the total change in the quantity of solute round the anode and cathode measured. As explained in the first part of this paper, the value of this change in gramme-equivalents, divided by the number of gramme-equivalents of hydrogen evolved at the cathode, gives us the migration constant for the solution.

The great advantage of sulphuric acid for these experiments is that no uncertain reactions take place at either anode or cathode. Measurements of the change in concentration at the two electrodes are thus equally trustworthy, and each serves as a check on the other.

The solutions used were made up with water having a specific conductivity of from $0{\cdot}9\times10^{-6}$ to $1{\cdot}0\times10^{-6}$ reciprocal ohm at 18° C. Ordinary distilled water was redistilled in a Jena glass apparatus with a trace of sulphuric acid and of potassium bichromate. A stock solution of sulphuric acid (of strength about 0·1 normal) was prepared from some pure 50 per cent sulphuric acid supplied by Messrs. Baird and Tatlock. The conductivity of this solution was measured, and its exact strength thence determined by means of Kohlrausch's tables. Dilute solutions were obtained from this by taking a weighed quantity and adding the requisite amount of water.

After the passage of the current through the migration cell, the strengths of the solutions in the anode and cathode regions were determined by measuring their conductivities. For this purpose it is necessary to know how the conductivity varies with concentration. The relation between the two has been determined by several observers, and the results plotted in the form of a curve showing the ratio of the equivalent conductivity to the cube root of the concentration. It was thought advisable, however, to have such a curve for the actual solution under observation. Since the solutions were all made up gravimetrically from the original one, we have at once a means of finding the concentration (in terms of that of the original). The conductivity was also measured. Each solution made up thus gave a point on the curve. From the curve so drawn, calculations were made for determining the strengths of the two solutions after the passage of the current. Slight effects due to the solution of impurities during the course of the experiment would give too small a value for the change in concentration on the one side, and too large a value on the other; taking the mean therefore tends to eliminate these effects; agreement between the changes on the two sides is a sure test that such errors have not entered to vitiate the results. This is a very important consideration, for with such weak solutions as were used, fouling, not only by the solution of solid matter from the surface of the vessel, but also by the absorption of gases such as ammonia from the air, is very likely to occur. Without such a check it would be dangerous to draw any conclusions as to the value of the migration constant for solutions weaker than 0·0001 normal, whereas, with the help of this check, successful experiments were conducted with solutions as weak as 0·00005 normal.

2. *Apparatus.*

1. *Conductivity Cells.*—Two cells were used in the course of the experiments for the purpose of measuring the conductivity, one for the two strongest solutions (0·1 and 0·01 N), and the other for solutions of strength 0·001 N and under.

The electrodes in both cells were of platinum, but in the case of the cell used for the stronger solutions (Fig. 2), they were coated with platinum-black, while in the other cell (Fig. 3), after platinisation, they had been heated to redness before being sealed into the glass. In the second cell, contact with the electrodes was made by means of mercury poured into the side tubes. The centre tube leading to the cell-chamber was closed with a glass cap, by the use of which, it was found, fouling from the atmosphere was prevented completely.

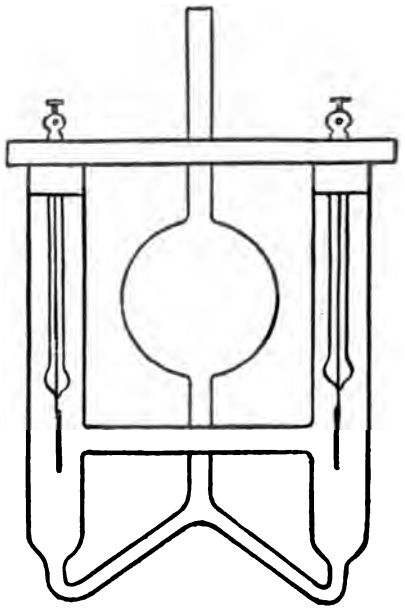

Fig. 2.

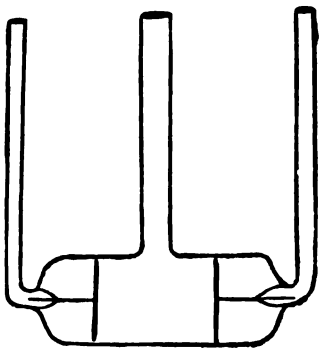

Fig. 3.

The cell-constant for each cell was determined by means of a standard potassium chloride solution made up from the carefully prepared, pure, crystallised salt. Knowing the concentration of this solution, we can calculate its conductivity (in reciprocal ohms) from the tables compiled by Kohlrausch.

The resistance in each case was determined by the method of Wheatstone's bridge. By the use of the commutator described in a paper by one of the authors (*Phil. Trans.*, 1900, A, cclix., 321), an alternating current was obtained, and at the same time a galvanometer used for testing the balance. The commutator was driven at the requisite speed by means of a small electric motor. In all

* A Paper read before the Royal Society, June 18, 1908.

measurements of the resistance the cell was placed in a bath of water kept well stirred and maintained at a temperature of 18° C., as shown by a Beckmann thermometer reading to hundredths of a degree. The temperature of the solution was regarded as constant and uniform when the measured resistance remained unaltered with time.

2. *The Migration Cell.*—This has been described in the first part of the paper. It may further be noticed here that by placing a pair of electrodes on each side, it was possible to measure the resistances of both portions of the liquid immediately before and immediately after the passage of the current, without transference to other conductivity cells. An almost certain cause of fouling was thus avoided. This device could be employed only for the weaker solutions (0·0002 N and under), as the resistances in the case of stronger solutions would be too small to be measured accurately with these electrodes. But with the stronger solutions, any possible fouling on transfer would be inappreciable.

3. *Galvanometer.*—The current through the migration cell was measured by means of a Nalder's moving-coil galvanometer, which, with the shunts used in the experiment, was previously standardised. This was done in the following way:—A current from a single storage cell (with a suitable resistance in series) was passed through a milliampère- (or volt-) meter, of resistance exactly 1 ohm. The terminals of the galvanometer were connected with the terminals of the ampère-meter through convenient resistances. From the reading of the ampère-meter and a knowledge of the resistance in the galvanometer circuit, we can calculate the current through the galvanometer. The deflections of the suspended coil of the galvanometer (observed by the ordinary reflection method) were noted simultaneously.

3. *Experimental Details.*

The method of procedure for solutions of strengths 0·0005 normal and over, differed slightly from that for weaker solutions. In the case of the stronger solutions the conductivity was too great for the resistance to be measured by means of the electrodes in the migration cell. Hence, the liquid had to be transferred to a conductivity cell. The conductivity of the original solution was first determined in one of these cells, in order to obtain a point on the equivalent conductivity curve. Some of the solution was then poured into the migration cell until the connecting tube was completely filled. The current (supplied by a hundred small storage cells) was passed through the apparatus for a time (generally ten to fifteen minutes) sufficient to produce on the average a 10 per cent change in the strength of the solutions; that is to say, until 10 per cent of the sulphate ions on the cathode side had migrated to the anode region. When the current had been stopped, the air pressure in the inverted V-tube was raised until the liquid had been forced to the ends of the tube, thus separating the anode and cathode portions. Each portion was then well stirred, and afterwards transferred by means of a syphon to a weighed flask, the syphon and flask having been carefully rinsed with pure water, and dried, before using. After the weight had been taken the solution was poured into the conductivity cell, and its resistance measured.

For solutions weaker than 0·0005 normal, the initial conductivity was determined in the conductivity cell as before. After the solution had been poured into the migration cell, the air pressure in the inverted V-tube was raised until the liquid had descended to the ends of the tube. The resistance on each side was measured, the solution being well stirred, and the temperature maintained at 18° C. After the air pressure had been released, and the liquid had risen to its former level, the current was passed through for the required time. The anode and cathode portions were again separated, as at the beginning of the experiment, and the resistances measured. Finally, the two portions were separately drawn off by means of a syphon, and weighed. The final conductivities are easily calculated, from the fact that they bear to the initial conductivity the ratios of the resistances (measured in the migration cell) at the beginning, to those at the end, of the experiment.

In calculating the conductivity of the sulphuric acid, the conductivity of the solvent was deducted from that of the solution in every case.

4. *Experimental Results.*

The following example shows the complete method of calculation for one of the experiments:—

Solution 20.

Original strength (as made up from a standard solution) .. .. $1{\cdot}0140 \times 10^{-7}$ grm.-equiv. per cc. ($=m_0$)

Resistance in conductivity cell .. .. 12916 ohms

Cell constant .. .. .. 0·4532

Conductivity of solution .. $\frac{0{\cdot}4532}{12916} = 3{\cdot}509 \times 10^{-5}$ recip. ohm

Conductivity of water (solvent) .. .. $0{\cdot}103 \times 10^{-5}$ „

Conductivity of acid (solute) .. .. $3{\cdot}406 \times 10^{-5}$ „ ($=k_0$)

(Hence $k_0/m_0 = 336{\cdot}0$, $\sqrt[3]{m_0} = 0{\cdot}0466$).

	Anode region.	Cathode region.
Resistances in migration cell, before	4825 ohms	5012 ohms
Resistances in migration cell, after	4276 „	5716 „
Weights of solutions ..	53·74 grms.	55·56 grms.

Calculations.

Anode side—

Conductivity after experiment .. .. $3{\cdot}509 \times 10^{-5} \times \frac{4825}{4276} = 3{\cdot}959 \times 10^{-5}$ recip. ohm

Conductivity of water .. 0·103

Conductivity of acid (solute) .. .. $3{\cdot}856 \times 10^{-5}$ ($=k_a$)

Concentration from curve $\left(\frac{k_a}{(k/m)}\right)$ .. .. .. $\frac{3{\cdot}856 \times 10^{-5}}{339{\cdot}0} = 1{\cdot}1377 \times 10^{-7}$ grm. - equiv. per cc.

Increase in concentration $0{\cdot}1237 \times 10^{-7}$

Total increase in quantity of acid .. .. $0{\cdot}1237 \times 10^{-7} \times 53{\cdot}74 = 0{\cdot}665 \times 10^{-6}$ grm. equiv.,

Cathode side—

Conductivity after experiment .. .. $3{\cdot}509 \times 10^{-} \times \frac{5012}{5716} = 3{\cdot}077 \times 10^{-}$ recip. ohm

Conductivity of water .. 0·103

Conductivity of acid (solute) .. .. $2{\cdot}974 \times 10^{-5}$ „ ($=k_c$)

Concentration from curve $\left(\frac{k_c}{(k/m)}\right)$ .. .. .. $\frac{2{\cdot}974 \times 10^{-}}{332{\cdot}0} = 0{\cdot}8960 \times 10^{-7}$ grm. - equiv. per cc.

Decrease in concentration $0{\cdot}1180 \times 10^{-7}$

Total increase in quantity of acid .. .. $0{\cdot}1180 \times 10^{-7} \times 55{\cdot}56 = 0{\cdot}655 \times 10^{-6}$ grm.-equiv.

Mean of change for two sides .. .. $0{\cdot}660 \times 10^{-6}$ „

Current through cell .. $4{\cdot}53 \times 10^{-4}$ ampère

Flowed for 12 mins. .. = 720 secs.

Hence the number of grm.-equiv. of hydrogen liberated $= 4{\cdot}53 \times 10^{-4} \times 720 \times 1{\cdot}036 \times 10^{-5} = 3{\cdot}38 \times 10^{-6}$.

Migration constant or transport ratio .. $= \frac{0{\cdot}660 \times 10^{-6}}{3{\cdot}38 \times 10^{-6}} = 0{\cdot}195$.

(To be continued).

NOTICES OF BOOKS.

Chemie und Biologie der Pflanzlichen Sekrete. ("The Chemistry and Biology of the Secretions of Plants"). By A. Tschirch. Leipzig: Akademische Verlagsgesellschaft. 1908.

The author of this lecture, which was delivered before the Swiss Chemical Society, has made his name widely known by his researches on the application of the methods and principles of organic chemistry to biological problems, and the reprint gives a clear summary of our present knowledge of the secretions of plants. The word secretion is interpreted in its narrower sense, being taken to include resins and ethereal oils only, and of these the former are more particularly considered. The history of their investigation is discussed in an impartial and broad-minded spirit, and the doctrine of their formation without the collaboration of the plasma, in a special membrane layer, is put forward and explained. An interesting account is given of the author's own work on the isolation of some of the important resins, and the decomposition of them into their constituents, and the lecture concludes with a short discussion of the theory that the formation of plant secretions is due to the action of enzymes.

CORRESPONDENCE.

POTENTIAL ENERGY OF THE ELEMENTS.

To the Editor of the Chemical News.

Sir,—Since the publication of my paper on the "Potential Energy of the Elements" (Chemical News, xcvii., 302), my attention has been drawn to a recently published correction of a factor, based on more extended research that affects the values I have given for the potentials of the members of the helium family of elements. I have accordingly re-calculated them with this correction as follows:—

He	134487 K
Na	23338 „
Ca	13415 „
Sr	6141 „
Ba	2516 „

—I am, &c.,

Daniel J. Rankin.

August 21, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Academie des Sciences. Vol. cxlvii., No. 3, July 20, 1908.

Hydrates of Strontium and Barium Oxides.—M. de Forcrand.—In a vacuum at about +10° the hydrate, $SrO + 9H_2O$, is very slowly dehydrated. It yields $SrO + 2H_2O$ after a week, and this substance continues to lose water for a month, the final product being $SrO + H_2O$. To get the anhydrous oxide this must be heated in dry hydrogen to 850° for some time. These results are obtained much more quickly when $SrO + 9H_2O$ is heated to 45—50° in a current of dry hydrogen, although the temperature has to be raised to 850° to ensure complete dehydration. Between the monohydrate and the anhydrous oxide intermediate substances, *e.g.*, $SrO + 0{\cdot}22H_2O$, $SrO + 0{\cdot}06H_2O$, appear to be formed. With barium oxide the phenomena exhibited are similar, but there is no evidence of the existence of the intermediate substances. Thus it is more difficult to pass from $SrO + H_2O$ to SrO than from $BaO + H_2O$ to BaO. It seems probable that the formulæ of the anhydrous oxides are really $(SrO)_x$ and $(BaO)_x$, instead of SrO and BaO, and there is reason to believe that, in the case of oxides of polyvalent metals at any rate, the progressive condensations with partial dehydration are produced by a mechanism analogous to that of the formation of anhydrides and ethers or hydrates of carbon in organic chemistry.

Researches on the Ketodiacids.—E. E. Blaise and H. Gault.—The saponification of oxalsuccinic ether by hydrochloric acid in the cold, followed by boiling to eliminate the β-ketoacid function, yields α-ketoglutaric acid,—

$$CO_2C_2H_5{-}CO \atop CO_2C_2H_5{>}\overset{\alpha}{C}H{-}\overset{\alpha'}{C}H_2{-}CO_2C_2H_5 \rightarrow$$

$$\rightarrow CO_2H{-}\overset{\alpha}{C}O{-}\overset{\beta}{C}H_2{-}\overset{\alpha'}{C}H_2{-}CO_2H.$$

Excellent yields of α′-methyl-α-oxalsuccinic ether can be obtained by condensing ethyl pyrotartrate with ethyl oxalate by means of sodium ethylate. The condensation of dioxalsuccinic ether with concentrated hydrochloric acid at the ordinary temperature gives an acid compound, from which, by boiling, the author has prepared isopyromucic acid, which must be β-oxy-α-pyrone. The acid compound is therefore the α′-carboxyl derivative of β-oxy-α-pyrone.

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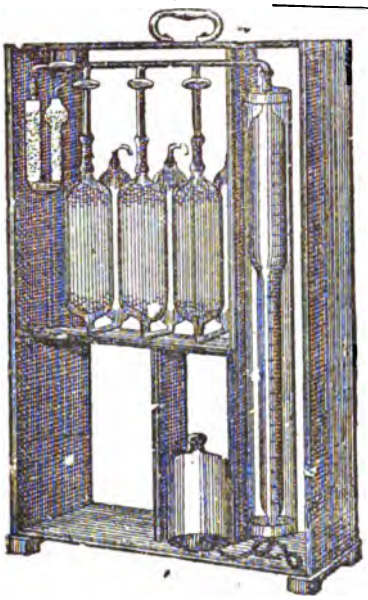

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THE CHEMICAL NEWS.

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ADDRESS TO THE CHEMICAL SECTION OF THE BRITISH ASSOCIATION.

DUBLIN, 1908.

By Prof. F. S. KIPPING, D.Sc., Ph.D., F.R.S., President of the Section.

On taking the Chair of this Section my first duty is to express my personal thanks to the Council of the British Association for having chosen me to fill the position of honour.

At this meeting the Association is enjoying, not for the first, but for the fourth time, the generous and genial hospitality of the citizens of Dublin; it is my privilege, on behalf of all the members of this Section, to tender our cordial thanks to our hosts for giving us this opportunity of meeting again in the capital of Ireland.

During the past few months we have read in the daily journals—and we sincerely hope it may be true—that there are signs of the commencement of a great development of the resources of this Island; as such a desirable event must be closely connected with, and, indeed, may even be dependent on, the vitality of the chemical industries of the country, the moment seems opportune for the consideration of a subject which has a direct bearing on both commerce and chemistry.

Although this Section is chiefly occupied with matters relating to pure science, the discussion of industrial questions is also regarded as one of its important functions; it does not attempt to distinguish pure from applied chemistry, and any problem which concerns either is deemed worthy of its attention.

From this point of view I propose to consider whether any steps can be taken to place the chemical industries of the United Kingdom of Great Britain and Ireland in a more prominent position than that which they now occupy in the world of commerce.

The subject is now new; it has been dealt with by many, but principally by those more directly interested—prominent members of the Society of Chemical Industry, who are far better qualified to express opinions on commercial matters than am I. It is perhaps presumption on my part to attempt to add anything to what has been said by such leaders of industrial chemistry, but I propose to deal with the subject from a very different standpoint—namely, from that of the teacher in the class-room and laboratory. Even if I fail to make a single suggestion of immediate practical value, the question is one of such magnitude and so many-sided that I feel justified in bringing it under the notice of this Section. It is not merely a matter of money, of a few millions or of a few tens of millions sterling. There are few branches of industry to which chemistry, in one way or another, is not of supreme importance. Whether we look to the great shipbuilding interests, dependent on the progress of metallurgy; to our cotton and linen trades, where cellulose reigns supreme; to our dye-houses or to our breweries, or to any other industry, great or small, there do we find problems in chemistry awaiting solution, and the nation which solves them will not only progress in civilisation and contentment, but will also justly claim to have taken a leading part in the advancement of science.

It is not then in any grudging spirit of envy that we approach this question; recognising the splendid work of men of other countries, rejoicing in the services which they have rendered to the world at large, our only desire is not to lag behind in the general intellectual and industrial advance of nations.

It is unnecessary to trouble you with any detailed comparison of the position which we occupy to-day with that which we have taken in the past. The fiftieth anniversary of the epoch-making discovery of mauve was held only two years ago, and the proceedings are still fresh in our recollection; the pæans of congratulation addressed to the discoverer (now, alas! no longer with us) were marred by a plaintive note, a note of lamentation over our lost industry, the manufacture of dyes. The jubilee of the founder of the colour industry in this country was also the occasion for pronouncing its funeral oration. If this were the full extent of our loss we might bear it with equanimity; but it is not so much what has already gone as what is going and what may go that are matters of such deep concern. Those who doubt the seriousness of our condition may find statistical evidence, more than sufficient to convince them, in the technical journals and in the Board of Trade reports of recent years.

The facts there disclosed show that in the manufacture of "fine chemicals," including perfumes, alkaloids, and crude coal-tar products, as well as dyes, the decadence of our industry is far advanced; in the case of heavy chemicals our position, perhaps, is not quite so serious at the present moment, but the future is dark and threatening. Chemical industries are so intimately connected and dependent on one another, that the fate of one may determine the fate of all; the by-product of one process is often the raw material of another. Who, then, can deny that the patience, perseverance, and high scientific skill, which have built up the colour industry abroad, if applied, as they have been and are being applied, to the manufacture of heavy chemicals, will not soon defy all competition from less progressive countries?

Such a possibility is full of national danger. It has been pointed out—and the prophecy cannot be regarded as unduly pessimistic—that from present indications a time will arrive when we shall be dependent on outside sources, not only for our food-supply, but also for our means of self-defence. When nitrates are exhausted, when nitric acid and ammonia are prepared from the components of the atmosphere, when all chemical industries have been so highly developed abroad that they have completely vanished from these Islands, and when their loss has reacted on all our other important industries, then, indeed, shall we feel the pinch of poverty; then, indeed, must we submit to national decay.

Is it possible to remedy the present unsatisfactory state of affairs, and to guard against an ominous future?

During the Perkin Jubilee celebrations Professor Carl Duisberg answered this question, in so far as it concerns the coal-tar colour industry, by an uncompromising negative. In an able and interesting speech he pointed out that, although the Briton is in general a practical man, he is lacking in patience, in the power of waiting for success; he expects to be compensated in hard cash, and at once, for his work or for his capital outlay. The German, on the other hand, is primarily a theorist possessing endless patience, and works without any immediate prospect of pecuniary reward; he has now learnt to be practical as well, but not at the expense of his ideals. It is to this happy combination of qualities that Professor Duisberg ascribes the success of his countrymen in the coal-tar colour industry—a success which he considers we are powerless to emulate, with which it would be futile for us to try and compete.

With this view that our chemical industries must submit to gradual extinction, even when it is held by so high an authority, we cannot and must not agree; if one nation can learn to be practical, we—the four nations of these islands—one or all, can learn to be plodding and patient, and to appreciate the importance of theory. We may be encouraged in our efforts to do so by the opinions of others, countrymen of Professor Duisberg, eminent in pure or applied science. Professor Ostwald, discussing this subject, said that he was sure the difficulties were considerable only in the beginning (*Journ. Soc. Chem. Ind.*, 1906,

1019), while Professor Lunge, in an address to the Royal Institution (March 15, 1907), made use of the following words:—"Seeing that in pure science the people of Great Britain haver never lagged behind any other nation, and that, on the contrary, the land of Newton and Faraday has been a beacon to all others at more than one epoch, there is absolutely no valid reason why she should now, or at any other time, be behind any other in the combination of science with practice."

Here, indeed, is encouragement, and from one who has had ample opportunity for studying the conditions which obtain in this country. Surely, therefore, we ought to have some confidence in ourselves and try our best to regain a strong and healthy position rather than fold our hands in a spirit of hopeless resignation.

The new Patent Act which came into force this year, and for which the country is so much indebted to the strenuous advocacy of Mr. Levinstein and Sir Joseph Lawrence, seems to many to have inaugurated a new era, and to have removed one of the principal causes of the decline of our chemical industries; if this be so, it is all the more important that the representatives of chemical science should be ready and willing to join hands with the manufacturers in order to assist in the process of regeneration.

The principal changes which have been introduced by the new law are, of course, familiar to all. The most important one, which came into operation on August 28 last, is that which requires that the article or process which is protected by the patent must be manufactured or carried on to an adequate extent in the United Kingdom after the expiration of four years from the date of the patent. If this condition is not fulfilled, any person may apply for the revocation of the patent.

Some of the results of this amendment, and some indications of the great industrial changes which it will bring about, are already obvious. Foreign firms or individuals who hold British patents and who have not sufficient capital to work them in this country, or who do not think they are worth working here, are attempting to sell their British patent rights. Others are building or buying works in Great Britain, and it has been estimated that in the immediate future a sum of at least £25,000,000 of foreign capital will have been thus invested in order to comply with the new law.

We need not stop to consider the economic effects of this transfer of capital on the general trade of this country, but we may well pause a moment in order to try and forecast the consequence of these new conditions in so far as they concern our chemical industries.

The prospective establishment of branches of two of the largest German chemical works at Ellesmere Port and at Port Sunlight respectively are already matters of common knowledge, and it may be presumed that these firms will avail themselves to a large extent of British labour. If this be the case, and if they are successful—as they, no doubt, will be—the complaint that the inferior technical education of our artisans is responsible for our lack of success will thereby be proved to be groundless. Even if we admit that at the present time the British workman is an inferior operative in a chemical works, and only capable of undertaking the less-skilled labour, these firms will gradually raise a considerable number of trained men who will be ready to undertake more responsible duties under our own manufacturers when the good time comes; a school for chemical operatives will be created in our midst, and, as in the past, we shall reap the benefit of knowledge and experience brought to our shores. It also seems reasonable to expect that, as is the case abroad, these works will be equipped with laboratories and staffed by chemists, although possibly only so far as is necessary for routine work. Many of these chemists may settle permanently in our midst, become members of our Chemical Society and Society of Chemical Industry, and thus infuse us with their patience and perseverance. It is not beyond the bounds of possibility that these great firms may even employ British chemists in their works, if we can supply men sufficiently well trained to be of value. On the other hand, as experience seems to have shown that industrial chemistry cannot succeed with imported scientific labour, it is not very probable that many posts in the laboratory will be filled by our countrymen, who, in this connection, must be regarded as foreigners.

Now, at the present time most chemical products can be manufactured more cheaply abroad than here, otherwise we should not have any reason to consider our position. Dr. Duisberg told us that even when an important firm in England had a licence to work all the British patents of two of the largest German colour works, merely paying for the privilege a small percentage of the net profits, it failed to take any advantage of the opportunity. If, then, in this free-trade kingdom production is cheaper than abroad, the foreign firms which have branches here will be in a position superior to that which they now occupy in their own countries. If, on the other hand, owing to inefficient labour, higher wages, freights, and other economic conditions, production is more costly, the superior efficiency and scientific organisation of these foreign firms will nevertheless enable them to command our home market with the goods made here, and to cut us out in the world market, as they do now, with those made abroad.

The conclusion which thus seems forced upon us is, that, although the new Patent Act will prove to be of great value in many respects, it will do little to foster British chemical trade and the development of British chemistry; it places us on an equality with other countries as regards patent rights, and thus remedies an outstanding grievance; but, unless we have something to patent, this equality will be valueless, and our chemical industries will continue to decline, possibly more rapidly than heretofore.

Let us therefore pass in review the other causes which have been suggested as contributory to our failure; after eliminating those connected with freights and tariffs, and with the alleged supineness of the Government in assisting industry, matters which may be left to the manufacturers to deal with, there still remain several which are well within the purview of this Section.

These are:—(1) The unsatisfactory condition of secondary education; (2) the nature of the training which is given to chemists in our universities and other institutions; (3) the insufficiency of the time and money devoted to research in the manufacturing industries; (4) the lack of co-operation between manufacturers and men of science.

There are some who believe that the first of these is the primary, if not the sole, cause of our weakness; that if our secondary education were placed on a sound basis all the other evils would disappear of their own accord; that a steady and broad stream of well-trained boys from the secondary schools would afford ample material from which good chemists could be fashioned in the universities and colleges; that these trained chemists would be greedily seized by the manufacturers, whose minds had been widened by improved educational methods; and once installed in the works these chemists would have no difficulty in persuading their employers to spend time and money on research work in co-operation with the leaders of science.

Whether such desirable and far-reaching results would, in fact, follow if our system of secondary education were very much improved it is impossible to predicate; but there is no doubt that at the present time we are moving in an exactly opposite direction.

The shadow of the cypress rests upon our chemical trade, and manufacturers do not see their way to employ chemists; students are not attracted to chemistry as a profession because there are so few openings; without an ample and increasing supply of such students chemical industry must continue to decline, and as a necessary consequence the development of pure chemistry is cramped and hindered to a far greater extent than is generally realised.

In a Presidential Address to the Chemical Society last year Professor Meldola discussed the position and prospects

of chemical research in Great Britain, and in view of the importance of the subject and the able manner in which it had been treated, the Council of the Society ordered the publication of five thousand copies of his Address for distribution among the members of various public bodies. We were told in this Address that many of our universities are distinct failures as centres of chemical research, and that the output of original work from our colleges, polytechnics, and similar institutions is emphatically not representative of the productive power of the teachers there employed. The causes of the failure of our universities were only lightly touched upon, and I propose to refer to them later; but in the case of our other institutions they were more fully discussed. May I venture to draw attention to one cause, which I believe is by far the most effective drag on research in the vast majority of such institutions not of university rank? It is simply the lack of those more advanced students who, while gaining valuable experience in the methods of research, would also render useful assistance to their teacher. The governing body of the institution may not realise the importance of research; the Principal, as, alas! is sometimes the case, may throw cold water on such work; the teacher may be overburdened with routine duties, and he may be most inadequately remunerated; if, however, the research spirit is strong within him, he would overcome all these difficulties were there any prospect whatsoever of success; but what chance has he when he must do everything himself, even to washing out his own test-tubes? Provide him with a few advanced students, and he would doubtless find time to undertake the necessary pioneer research work, which would then be extended and developed with their assistance.

It might be suggested that an efficient and enthusiastic man would soon attract a number of research students. This, no doubt, is true as regards the universities, but it must be remembered that a polytechnic or other institution which does not grant degrees can hardly expect to compete with a university as a centre for research; all those students who intend to undergo a so-called "complete" course of study—that is to say, all who are likely to become capable of undertaking research work—naturally proceed to one of the degree-giving universities. There are not enough students to go round, to satisfy the research requirements of the teachers, and the principal reason is—the limited demand for trained chemists on the part of the manufacturers.

Even of the small number of those who leave our teaching institutions fairly well trained in research, how many have a chance of passing into works and directly advancing applied science? A very small proportion indeed. Most of the better ones drift into other posts, become demonstrators, emigrate—anything rather than wait on with the prospect of accepting as works-chemist a salary which, meagre though it be, may be stopped altogether if dividends are low.

With whom rests the responsibility for this state of affairs? Is it with the teachers, and, if so, is it because they are incapable of training chemists or because their system is at fault?

To answer this question it is necessary, in the first place, to arrive at some conclusion as to the kind of training which is required for the future works-chemist. On consulting the opinions of the manufacturers it would seem that they attach great importance to what is called the "practical side"; they believe that, in addition to a knowledge of theoretical chemistry, the prospective works-chemist should also have some acquaintance with engineering, should understand the apparatus and machinery used in the particular manufacturing operations with which he is going to deal, and should have had practical experience in working the given process. It is from this point of view that we build and equip large technological chemistry departments, such as those in the Universities of Birmingham and Leeds, and in the Manchester Municipal School of Technology, departments fitted up with complete apparatus and machinery for carrying out operations on a miniature manufacturing scale.

The arguments in favour of this view, that it is a hybrid chemist-engineer who is required in a chemical works, seem to me to be fundamentally unsound, and the kind of training suggested by them for the works-chemist can only result in the production of a sort of combined analytical machine and foreman. A two or three years' course of science, followed by one year's practical work in the dye-house, in paper-making, or in some other technological department, is quite inadequate if the student trained in this way be expected to do anything beyond routine analytical work and supervision.

We cannot possibly expect such a poorly trained Jack-of-all-trades to run a chemical works successfully in the face of competition directed by a large staff of scientific experts in chemistry and in engineering. It is no use spending immense sums of money on expensive machinery of the newest type in order that the works-chemist may be able to tell his future employer that the machinery used in his employer's works is completely out of date. In the course of time, moreover, unless expenditure is practically unlimited, the reverse conditions will obtain, and the technological department of the university or other institution will become more of the nature of a museum of antiquities. The great cost of the upkeep and of the working of such plant is also a very serious matter; is it possible to believe that the educational results of running, say, a large puddling furnace, such as is fitted up in Birmingham University, are in any way commensurate with their cost? The conditions in a chemical works cannot be successfully imitated in a university or polytechnic; attempts to do so can only lead to mistaken conclusions, and thus have the effect of rendering the works-chemist quite helpless when he passes from the elegant models of his educational apparatus to the work-aday appliances of the manufactory.

Here, it seems to me, we touch the bed-rock of our trouble. The state of our chemical industries must be attributed to the erroneous views which have been and still are held as to the functions, and consequently as to the training, of a works-chemist. We have failed to realise that industrial chemistry must be based on a foundation of continuous and arduous research work. In the past we have sent out from our universities and other institutions students who, no doubt, were qualified to undertake routine analytical work, but the great majority of whom knew nothing of the methods of research. We are doing the same to-day. Just when a student has reached a stage at which his specialised scientific training should begin his course is finished, and whether he has been to a university or to a polytechnic matters little; he joins the band of those who subsist on but who do nothing to advance chemical industry. He enters a works; the manufacturer does not realise exactly what his chemist ought to do, but he expects some immediate results, and in consequence is generally disappointed; the lack of success of the chemist is put down to his ignorance of practical matters, and there is an outcry for technical education; science is most unjustly discredited, and any suggestion of spending money on research work is scouted as a mere waste.

The consequence is that there is a scientific problem which intimately concerns all the members of some large industry what course do they adopt? Through their trade journal, and as an association representing a total capital of which I should not like to hazard a guess, they offer a bronze or possibly a silver medal, or may even offer the extravagant sum of £20, to the happy person who will provide them with a solution. It is difficult to imagine the class of solvers to whom these princely rewards may appeal, more difficult still to believe that any useful result can be attained, and it is almost incredible that such methods should be adopted by any influential industrial organisation. This way of attempting to get research work "on the cheap" is certainly not unknown even in

more enlightened countries, but that is hardly a sufficient justification for its employment.

Contrast these methods with those adopted by the Badische Anilin- und Soda-Fabrik, and Meister, Lucius, and Brüning in their attempts to solve the problem of the commercial synthesis of indigo. Could there be a greater antithesis? If five thousand copies of Brunck's Paper on this subject (*Ber.*, 1900, [1], lxxi.) could be circulated among the manufacturers of this country—a task which might be fittingly undertaken by the Society of Chemical Industry—the study of the truly magnificent results attained by the systematic application of pure science, and of the indisputable evidence of their commercial value, might prove an object-lesson far more effective than argument for the accomplishment of a sorely needed reform.

Now if we are to meet successfully the very formidable scientific and commercial organisation opposed to us in chemical industry, we must perforce adopt the methods of our competitors; not only must we learn patience and perseverance, but we must also call to our aid the best brain-power available. We must recognise clearly that the scientific works-chemist, the only man who is likely to make discoveries of commercial value, must be thoroughly trained in the methods of research by those best qualified to do so, and we must not imagine that when he enters the works he should or could immediately become an engineer and a commercial expert; his place is in the research laboratory. The practical man—that is to say, the man who has a thorough and useful knowledge of some particular manufacturing process—must be trained under practical men in the works, and we must not imagine that a course of evening classes will convert him into an expert chemist. The ideal man who combines high scientific training and sound practical knowledge cannot be produced unless the period of his education is extended to half a life-time, and even then only through the co-operation of the chemistry teacher and the manufacturer.

Admitting the truth of these statements—and I do not think that they can be successfully controverted—we have now to consider what steps can be taken to provide these highly trained works-chemists, and to ensure for them a cordial reception on the part of the manufacturers.

The first fact which we have to bear in mind is that the great and rapid development of chemistry in recent times has lengthened the period which is required for the collegiate study of the subject. In order to acquire the necessary knowledge of facts and theory, and afterwards to devote even the minimum time to gaining experience in research methods, the future works-chemist must be prepared to continue at the university or other institution during at least five years. The course of study during the first three years might be on the lines now adopted by many of our universities for the B.Sc. pass examination, but to grant this degree in one or two subjects only, and then to call it an Honours degree, is in my opinion a serious mistake, as is also the admission of research work at this stage, both of which proceedings lead to far too early specialisation. The pass degree should be regarded merely as an indication of a sound general education in science, and the future works-chemist should then devote at least two years more to research and to special work in chemistry, on the results of which the Honours degree might be awarded. Every encouragement in the form of low fees, free admission, research scholarships, and so on, should be offered to such students, according to their merit and circumstances, in order that they may prolong their studies; the cost of these remissions or awards would not be very serious, and the money would be well spent. Teachers should then refuse to recommend, and manufacturers should refuse to employ, as a works-chemist, any student who had not passed through such a course satisfactorily, unless it was understood that he was only expected to undertake routine analysis, or work outside the research laboratory. By thus extending the period of training, and making research work compulsory as far as possible, a great deal would be gained; pure science would reap an immediate benefit from the investigations of the students—as has been the case abroad—and this stimulus would necessarily react on industrial chemistry; the manufacturers could be assured that they were being supplied with men of the right type; they would soon come to recognise that fact, and the demand for works-chemists would expand. In the laboratory of the works the manufacturer would then have the opportunity of gauging the capabilities and special leanings of every chemist on his staff. Those who were best fitted for directing operations in the works could be trained on the spot, as they could not possibly hope to be trained in any university or polytechnic; those who proved to be the best research chemists would, of course, remain in the laboratory working out scientific problems. Organisations of this kind could not fail to command success, and the opsonic curve of our chemical industries would soon begin to rise.

There is one institution, not a teaching body, which might greatly assist in this movement; I refer to the Institute of Chemistry of Great Britain and Ireland. This body desires and claims to represent chemistry, not only in these islands, but in all our dominions, and also to exercise some supervision or control over public appointments. It examines in chemistry and grants diplomas, and claims that its examinations are a test of practical ability rather than of theoretical knowledge. I have not a word to say against the character of these examinations, but to imagine that the Institute of Chemistry qualification is the hall-mark of a chemist is ridiculous. An average student can obtain the diploma after three, or at the very most four, years' work subsequent to matriculation, and more easily than the London B.Sc. (external). Here, again, it should be recognised that the present Institute of Chemistry qualification is only a step in the training of a chemist; the permission to present a thesis for the Associate examination should be withdrawn, and good research work should be insisted on in the case of all candidates for the Fellowship. It would then be possible to distinguish between those who are capable routine chemists and those who might be expected to advance pure and applied science. It is certainly a grave matter for an Institution entirely controlled by chemists to set such a bad example by ignoring the necessity of research work; if all our official chemical appointments and many of our posts in works are to be filled by men who have done no independent scientific work the results will be most serious; the research habit and the research method are not easily acquired without assistance, and therefore it is all the more important to make use of this assistance while it is within reach, and before the budding chemist begins to believe that he has nothing more to learn.

As a necessary corollary to making research compulsory in the training of works-chemists, all our important teaching institutions must afford ample opportunities for such work, and measures must be adopted to guard against that failure of some of our universities as centres of research which was pointed ont by Professor Meldola.

Such failure, whatever may be the contributory causes, must be principally due to the absence of sufficient interest in research work on the part of the professor, and it certainly seems surprising, at first sight, that in these days many such professors are to be found; but it must be remembered that, although by members of this Section research work is regarded as the highest and most important of all professorial duties, this is not always the view of those who make an appointment to a Chair.

In selecting a professor there are many other considerations which come into play: his ability as a teacher in the class-room and laboratory; his qualifications as a popular exponent of science; his power of organisation; his bearing towards his colleagues and his students—all these matters are of great and direct importance to a university, and it is not to be wondered at that a man highly qualified in these accessories may sometimes be chosen even though he may take no special interest in research work.

The results of such an appointment, however, cannot fail to be most prejudicial to the highest interests of the university and of the country; the chemistry department becomes a chemistry school, but not a school of chemistry.

Unfortunately, moreover, the results extend over a long period; this raises another question which certainly requires attention if we are to become more efficient.

It is far from my object to create any gratuitous insecurity of tenure in chairs of chemistry, but it is not desirable that in our teaching institutions the conditions of all appointments should include a superannuation clause? Not that a rigid age-limit should be introduced, but there should be a possibility of bringing about the retirement of those who for any reason can no longer adequately fulfil their duties. When, owing to the lapse of time, such retirement became necessary, the aged and honoured professor, pensioned by a grateful university, might still retain an intimate connection with its scientific life; as emeritus professor, with a research laboratory at his disposal, he might remain to advise and encourage his youthful successor even when the duties of teaching and the general supervision of a department had become too arduous.

It cannot be suggested that my remarks on this delicate topic are inspired by the impatience of youth or by freedom from personal consequences; the time when superannuation becomes desirable may arrive for one and all, and I have ventured to draw attention to the matter simply and solely because of its grave importance in connection with the subject of my Address. The country cannot afford to allow periods of inactivity or decadence in our seats of learning, and the interests of the individual must be subordinated to those of the nation.

Even if by adopting the above suggestions the training of our chemists is improved, and all our higher educational institutions become permanent and active centres of research, the manufacterers may still remain unresponsive, what can be done in other ways to bring about the active co-operation of pure and applied science?

The great proportion of the original work now done in this country, judging from the published records, is absolutely free from any utilitarian bias; the time, brain-power, and money devoted to this work are considerable, and the results from a scientific point of view eminently satisfactory. If even a fraction of the same skill and energy were brought to bear under proper conditions on problems of applied science, who can doubt but that the effect on our chemical industries would be one of vast importance? And yet it is the rarest possible occurrence to find any record of research work undertaken with a commercial object even in the natural home of such records, the *Journal of the Society of Chemical Industry*.

One reason for this may be that the discoveries made in the works-laboratories are not given to the world at large, but are quietly and lucratively applied in some secret manufacturing process. Another reason, unfortunately the more probable one, may be that nearly all the principal research workers are completeiy shut off from any industrial influences.

Now the worker in pure science, unaided by the advice of the manufacturer and business man, has little chance of solving any important technological problem, except as the result of accident; he has not the requisite acquaintance with commercial conditions, does not realise the enormous difference between operations on the laboratory and the manufacturing scales, or, if he does so, is unable to enter fully and with confidence into questions of fuel, labour, and so on, which often determine the success or otherwise of a process. Further, much of the research work of direct commercial value concerns methods for reducing the cost of processes already in operation, and demands an intimate practical knowledge of these processes.

It is obvious therefore that, even if all the research capacity of the country were henceforth devoted to purely technical matters, any great improvement in our industries could hardly be anticipated without the active co-operation of the manufacturers.

Now it has been stated (Levinstein, *Journ. Soc. Chem. Ind.*, 1903, 845) that the authorities of the Manchester Municipal School of Technology intend to undertake investigations for local manufacturers and merchants in connection with difficulties which may be met with in their works or business. This method of securing the interest and support of those engaged in applied chemistry may or may not be workable according to the conditions under which such co-operation is carried out. The staff and the laboratories of a university or polytechnic cannot be placed at the unrestrained and gratuitous disposal of any manufacturer who is in some trivial difficulty, nor can the choice of the subjects to be investigated be decided by the governing body. If, however, the arrangements, pecuniary and otherwise, are left entirely in the hands of those directly concerned, namely, the manufacturer and the responsible head of the chemistry department, the scheme should then prove exceedingly valuable, and should be adopted as widely as possible. It should be understood, and the fact might even be advertised by the governing body, that for purposes of research work in applied chemistry—but not, of course, for analytical work—the laboratory of the university, college, or polytechnic is, under certain conditions, at the service of the manufacturers; that although primarily and unswervingly devoted to work in pure science, such institutions recognise that, for their own interests, they must do all they can to assist chemical industries.

It might be thought that these conditions prevail at the present time and that any manufacturer, if he so choose, may consult the university staff on any problem in which he is interested. Possibly this is true to a limited extent, but in most institutions the members of the staff are restrained from undertaking any outside work; in others, such work may only be done with the sanction of the authorities.

These conditions, of course, are only laid down because the governing body believes that they safeguard the interests of the institution, and if it were shown that their enforcement is really contrary to those interests they would soon be abrogated. Many or all such authorities readily permit the members of their staff to undertake outside examination work because they consider this course to be to the advantage of their institution; but how incomparably more important is the object of gaining the confidence and support of the manufacturers.

Pray do not let it be imagined that this is some subtle scheme for increasing the pecuniary rewards of the teachers. I greatly fear that to many of those who are now engaged in research work the suggestion that they should give some attention to applied chemistry would be very distasteful, simply because it would involve an immediate encroachment on the time, already far too limited, which they are able to give to the immediate scientific problem which is one of their principal joys in life. To those who might have fears of this kind I would point out that there would soon be some compensation; once the co-operation of the manufacturers is secured, the demand for research chemists would expand, and the laboratories would be filled with students whose help in pure science would be invaluable.

The possible objection that the teaching staff would devote too much time to applied work and neglect other duties is one which could be left for the governing body to deal with unsparingly. If the institution took some percentage of all extraneous remuneration, or any similar arrangement were made, the funds thus provided could be used for increasing the staff of assistants and demonstrators—a most desirable reform in itself.

One of the greatest advantages of a working arrangement such as that here indicated would be that, like the method already suggested, it would lead to the evolution of what is otherwise almost unattainable—namely, men thoroughly trained in both science and practice. The research students of the teaching institution, engaged on a given problem for a manufacturer, would of course be allowed to study its practical aspects in the works; on the

other hand, works-chemists, with considerable practical experience, would be granted permission to proceed to the university laboratory, where they would study the problem with the assistance of the highest scientific knowledge, and acquire further training in the methods of research.

Combinations such as these could hardly fail to lead to valuable results, which would form the subject of patents; the monopolies thus acquired would place the manufacturers in a favourable position, and the revival of our chemical industries would follow in due course. There is nothing Utopian in this scheme, and there are no great initial difficulties to be overcome; it may be set in operation by the manufacturer, and possibly also, as will be indicated later, by the worker in pure science. Reading between the lines certain records which have recently appeared in the science journals and the patent lists, it may even be inferred that such arrangements are already in force in one of our large industrial centres.

There are other ways in which it might be possible to obtain the active co-operation of the manufacturers. Any individual or firm interested in a problem of applied science might be invited to found a temporary research scholarship at the university or other institution for the definite object of the particular problem in question. The maximum period during which such a scholarship would be tenable might be fixed beforehand, so that the financial liability of the founder would be limited and proportionate to the importance of the object in view. The holder of the scholarship might be nominated by the university, or by the founder and the university jointly, and suitable conditions would be drawn up to ensure the interests of the founder; he would of course have the benefit of all the results of the work, and would secure the patent rights of any new invention, subject possibly to the payment of a small percentage of the profits to the university and to the holder of the scholarship. During the tenure of the scholarship, the holder, and also the founder, would have the advantage of the scientific knowledge of the university; the scholarship holder would also be allowed to gain practical experience in the works, and, if successful, there is little doubt but that he would have the option of working the process on the large scale and of obtaining permanent employment under satisfactory conditions. After a given period the scientific results of the work would be published through the usual channels in the ordinary way.

This idea of applied research scholarships had taken shape in my mind when I happened to come across a book recently published in the United States, called "The Chemistry of Commerce," in which I found that a similar proposal had been made by the author, R. K. Duncan, Professor of Industrial Chemistry at the University of Kansas. The scheme is there worked out in some detail, and a form of legal agreement to be signed by the university authorities and by the founder of the "Industrial Fellowship" is suggested.

Thinking it would be of interest to know how the plan had worked out in practice, I wrote to Professor Duncan and received a reply a few weeks ago. He very courteously informed me that five industrial fellowships had already been established in his laboratories, that the agreements for two additional ones were being prepared, and that he might have obtained more but wished to proceed conservatively; also that he had no reason to doubt the entire practicability of the scheme, and that experience had shown that the terms of the agreement could be made more favourable to the university than those which were first drawn up. One of the new conditions is that the industrial fellowship holder shall give two hours a week gratuitous instruction in the work of the chemistry department—an arrangement which has proved to be of great inspirational value. The fellowships are tenable during two years and are of the value of 500 dols. or 1000 dols. per annum.

It is too soon to be able to form any opinion as to the commercial importance of the work carried out under this scheme, but it is obvious that the foundation of such scholarships for the study of general or special problems in applied chemistry is most desirable. One of their great advantages would be that they might be founded by those manufacturers who cannot afford permanently to engage a research chemist. Large and successful firms like the United Alkali Co., Brunner, Mond, & Co., and many others which can employ a staff of chemists, are of course eminently capable of managing their own affairs without outside assistance or advice, and it is only for those which are less prosperous that the foregoing suggestions are made.

The great benefits which are conferred on pure science by the open research scholarships at present available afford some indication of what might be done for industrial chemistry by the foundation of such scholarships in applied science. There are, no doubt, scattered over the country many men who possess originality and inventive talent, and who have practical experience in industrial operations, but who have not been sufficiently trained in science; if it were possible to attract this dormant talent by means of open scholarships it might be directed into proper channels instead of being allowed to run to waste.

It is easy to say how money might be spent advantageously, but very difficult to suggest how the funds for such open scholarships should be raised. An appeal to the manufacturers by this Association or by the Society of Chemical Industry might meet with some response, and it is also possible that public bodies might render assistance. If the Government of Bengal, under the spur of dire necessity, can subsidise research work on indigo, and if our county councils can offer scholarships for dairy work, and grants for experiments on turnip-growing, bee-keeping, and so on, our city and borough councils might award scholarships in applied chemistry for subjects of especial importance to the dominant trades of the district. By so doing they would be utilising to the best advantage the chemistry departments of our universities and polytechnics.

I noted a few moments ago that practically all the published research work of this country has no direct reference to any industrial problem; nevertheless the results of this work are often of such a character that they might be of considerable technological importance. New reactions are discovered; new or improved methods of preparing known compounds; new facts as to the conditions under which important general reactions occur; and, needless to add, a great many new compounds are prepared.

Now, abroad, all or nearly all such matters are protected by patents, generally taken out by some firm of manufacturers. To the uninitiated it seems absurd to think that there is money in the great majority of such patents, and yet it is obvious that the employment of this system must pay in the long run. Why should it not be adopted in this country—at any rate to a limited extent to start with?

If all those who are engaged in purely scientific research work would seriously consider the desirability of obtaining provisional protection for any discovery which they may make, and would then consult some manufacturer or industrial expert with whom the further development of the matter might be undertaken, there is reason to believe that in some cases at least the patent might prove to be a commercial success.

The examination of the therapeutic action of compounds discovered in our laboratories is also a possible means of assisting our chemical industries; the matter is not so trivial as it may seem; a monopoly in the manufacture of some valuable medicinal preparation would serve as a *point d'appui* from which more important operations could be undertaken.

Unfortunately the investigation of the physiological action of new preparations is a matter of some difficulty in this country, as it is to some extent connected with vivisection in the public mind; we may poison rats with impunity, and even create an organisation for their extermination, but we may not individually try the effect of a new compound on a rabbit.

In drawing this address to a conclusion I cannot but

feel that my suggestions may seem utterly inadequate to the attainment of those important results which are so greatly to be desired. If so, I can only plead that more drastic measures are hardly available, and that even under the most favourable circumstances improvement can take place only very slowly. Whatever differences of opinion may be held as to the details of any scheme for regaining our lost ground, the main lines seem to be clearly indicated. The workers in pure science must recognise that it is their duty to do all they can to promote the industrial welfare of their country; the manufacturers must concede the paramount importance of science and the impossibility of dispensing with its counsels. Guided by these principles and by a spirit of cordial co-operation, a sustained and strenuous effort on the part of the leaders of chemical industry and of chemical science can hardly fail to accomplish the end in view.

In elaborating this Address I have enjoyed the advantage of the criticisms and suggestions of my friend and relative Professor Perkin, F.R.S., to whom my sincere thanks are here expressed.

THE ISOMERISM OF ETHYL ACETO-ACETATE.

By R. H. McCREA.

In the general theory of the stereo-isomerism of unsaturated compounds the following types are met with :—

$$\text{I.}\quad \begin{matrix} R.C.H \\ \| \\ R_1.C.H \end{matrix} \qquad \text{II.}\quad \begin{matrix} R.C.H \\ \| \\ H.C.R_1 \end{matrix},$$

and it appears that by a slight extension of this the case of aceto-acetic ether may be covered. If we represent the formula for the enolic form, $CH_3.C(OH):CH.COOEt$, by the graphic method (II.) above,—

$$\begin{matrix} CH_3.C.OH \\ \| \\ H.C.CO_2C_2H_5 \end{matrix},$$

we see at once that the *cis*-configuration corresponding to this is—

$$\begin{matrix} CH_3.C.OH \\ \| \\ C_2H_5O_2C.C.H \end{matrix},$$

Now, in the case of the Beckmann transformation the following has been thought to be the probable course of the change (Bernthsen) :—

$$\begin{matrix} R.C.R' \\ \| \\ N.OH \end{matrix} \rightarrow \begin{matrix} R.C.OH \\ \| \\ N.R' \end{matrix} \rightarrow \begin{matrix} R.C=O \\ | \\ NHR \end{matrix}.$$

There does not seem, then, any difficulty in supposing the *cis*-modification unstable in the case of the ethyl ester, for—

$$\begin{matrix} CH_3.C.OH \\ \| \\ EtO_2C.C.H \end{matrix}$$

corresponds exactly with the hypothetical intermediate product above, and gives at once—

$$\begin{matrix} CH_3.C=O \\ EtO_2C.C<^{H}_{H} \end{matrix},$$

the ketonic form of the ester, by a change in the position of the double bond, the hydrogen atom of the hydroxyl changing over to the carbon atom, and the oxygen of the hydroxyl taking possession of the free affinity, just at the moment when the *cis*-position is attained. Afterwards, of course, there is free rotation about the single bond linking the carbon atoms.

Now, with phosphorus pentachloride β-chlorocrotonic acid is formed, $CH_3.CCl:CH.COOH$, which may be represented as either—

$$\begin{matrix} CH_3.C.Cl \\ \| \\ H.C.CO_2H \end{matrix} \quad \text{or} \quad \begin{matrix} CH_3.C.Cl \\ \| \\ HO_2C.C.H \end{matrix}.$$

We should here be led to expect that both modifications would be stable, for there is no element present in the chlorine radical to saturate two links of the carbon atom like the oxygen of the hydroxyl in the supposed unstable *cis*-modification of ethyl aceto-acetate. Nor is this expectation unsupported by facts, for the above chloro-compound yields isocrotonic acid, $CH_3.CH:CH.CO_2H$, under the action of sodium amalgam, which is taken as being stereo-isomeric with ordinary crotonic acid.

To the question as to which of the modifications, stable or unstable, of the ethyl ester should be regarded as the *trans*- or *cis*-form no answer is attempted. For convenience one form has been adopted above, but the other would serve the purpose equally well. It may be pointed out, however, that while the configurations of the crotonic acids have not been settled apparently, the *iso*-acid is formed from the chloro-*iso*-acid, which also gives rise to tetrolic acid, $CH_3.C:C.CO_2H$, by the loss of hydrogen chloride in presence of aqueous potash. Now, although the loss of a H and Cl atom does not necessarily imply that these are spatially near each other, if such were found to be the case, in this instance we could work backwards, thus,—

$$\begin{matrix} CH_3.C & Cl \\ \| & + \\ HO_2C.C & H \end{matrix} \rightarrow \begin{matrix} CH_3.C.Cl \\ \| \\ HO_2C.C.H \end{matrix} \rightarrow \begin{matrix} CH_3.C.OH \\ \| \\ HO_2C.C.H \end{matrix}.$$

Tetrolic acid → *iso*-Chloro acid → *cis*-Form.

The latter form in the case of the ester might perhaps be regarded as the stable hydroxy configuration.

Chesterfield, July 21, 1908.

THE ELECTROLYTIC PROPERTIES OF DILUTE SOLUTIONS OF SULPHURIC ACID.*

By W. C. D. WHETHAM, M.A. F.R.S., and H. H. PAINE, B.A., Trinity College, Cambridge.

(Continued from p. 120).

PART II.—*By* H. H. PAINE (*continued*).

TABLE II. contains the results of the whole series of experiments; its various columns will easily be understood from the preceding example. In deducing the mean of the several values for the migration constant at any particular concentration, those experiments were regarded as most trustworthy for which the calculated changes at anode and cathode were most nearly equal, and the values for the migration constant were weighted accordingly. The difference between the weighted mean and the actual one was always quite small.

The solution 0·0001 normal was the strongest for which the conductivity could be measured in the migration cell itself. Hence, for the solution 0·0002 normal in the case of which the anode and cathode portions had to be transferred to the conductivity cell, and for which the discrepancies between the changes on the two sides were rather large, the value obtained for the migration constant is not very trustworthy. Of the three experiments performed with this solution, that showing the best agreement between the measured changes at the anode and cathode is the one giving 0·188 as the migration constant.

It was found that the conductivities of solutions of the same strength made up with different specimens of distilled water were not the same. When the discrepancy was small, the procedure adopted in calculation was as follows :

* A Paper read before the Royal Society, June 18, 1908.

TABLE II.

I. No. of solution.	II. Initial concentration (grm.-equivalent per litre).	III. Total change in quantity of H_2SO_4 (grm.-equivalent). Anode.	IV. Cathode.	V. Mean.	VI. Grm.-equivalent of hydrogen liberated at cathode.	VII. Migration constant from Columns V. and VI.	VIII. Weighted mean.
1.	$1\cdot0465 \times 10^{-1}$	$4\cdot47 \times 10^{-4}$	$4\cdot67 \times 10^{-4}$	$4\cdot57 \times 10^{-4}$	$2\cdot485 \times 10^{-3}$	0·184	
2.	$1\cdot0465$ „	$5\cdot71$ „	$5\cdot83$ „	$5\cdot77$ „	$3\cdot079$ „	0·187	
							0·186
3.	$1\cdot0070 \times 10^{-3}$	$5\cdot82 \times 10^{-5}$	$6\cdot14 \times 10^{-5}$	$5\cdot98 \times 10^{-5}$	$3\cdot214 \times 10^{-4}$	0·186	
4.	$1\cdot0070$ „	$8\cdot65$ „	$8\cdot96$ „	$8\cdot80$ „	$4\cdot744$ „	0·1855	
							0·186
5.	$1\cdot0230 \times 10^{-2}$	$5\cdot50 \times 10^{-6}$	$5\cdot71 \times 10^{-6}$	$5\cdot60 \times 10^{-6}$	$2\cdot71 \times 10^{-5}$	0·206	
6.	$1\cdot0230$ „	$5\cdot49$ „	$5\cdot64$ „	$5\cdot56$ „	$2\cdot96$ „	0·188	
7.	$1\cdot0255$ „	$5\cdot49$ „	$5\cdot51$ „	$5\cdot50$ „	$2\cdot93$ „	0·188	
8.	$1\cdot0255$ „	$4\cdot09$ „	$4\cdot13$ „	$4\cdot11$ „	$2\cdot13$ „	0·193	
9.	$1\cdot0165$ „	$3\cdot37$ „	$3\cdot49$ „	$3\cdot43$ „	$1\cdot864$ „	0·184	
10.	$1\cdot0165$ „	$3\cdot52$ „	$3\cdot48$ „	$3\cdot50$ „	$1\cdot864$ „	0·188	
11.	$1\cdot0165$ „	$3\cdot90$ „	$3\cdot92$ „	$3\cdot91$ „	$2\cdot11$ „	0·185	
							0·189
12.	$0\cdot5133$ „	$2\cdot57$ „	$2\cdot59$ „	$2\cdot58$ „	$1\cdot402$ „	0·184	
13.	$0\cdot5133$ „	$3\cdot77$ „	$3\cdot83$ „	$3\cdot80$ „	$2\cdot05$ „	0·185	
14.	$0\cdot5081$ „	$2\cdot89$ „	$3\cdot02$ „	$2\cdot955$ „	$1\cdot582$ „	0·187	
							0·185
15.	$0\cdot20785$ „	$0\cdot644$ „	$0\cdot596$ „	$0\cdot620$ „	$3\cdot40 \times 10^{-6}$	0·182	
16.	$0\cdot20785$ „	$1\cdot008$ „	$0\cdot963$ „	$0\cdot985$ „	$5\cdot25$ „	0·188	
17.	$0\cdot20785$ „	$1\cdot100$ „	$0\cdot022$ „	$1\cdot061$ „	$5\cdot70$ „	0·184	
							0·1855
18.	$1\cdot0140 \times 10^{-4}$	$5\cdot765 \times 10^{-7}$	$5\cdot625 \times 10^{-7}$	$5\cdot695 \times 10^{-7}$	$2\cdot93$ „	0·195	
19.	$1\cdot0140$ „	$5\cdot15$ „	$5\cdot18$ „	$5\cdot165$ „	$2\cdot69$ „	0·192	
20.	$1\cdot0140$ „	$6\cdot65$ „	$6\cdot55$ „	$6\cdot60$ „	$3\cdot38$ „	0·195	
							0·194
21.	$0\cdot5440$ „	$3\cdot18$ „	$3\cdot25$ „	$3\cdot215$ „	$1\cdot566$ „	0·205	
22.	$0\cdot5440$ „	$3\cdot78$ „	$3\cdot78$ „	$3\cdot78$ „	$1\cdot827$ „	0·207	
23.	$0\cdot5405$ „	$2\cdot83$ „	$2\cdot83$ „	$2\cdot83$ „	$1\cdot392$ „	0·203	
24.	$0\cdot5405$ „	$3\cdot01$ „	$3\cdot08$ „	$3\cdot045$ „	$1\cdot522$ „	0·200	
25.	$0\cdot5832$ „	$2\cdot62$ „	$2\cdot56$ „	$2\cdot59$ „	$1\cdot359$ „	0·191	
26.	$0\cdot5832$ „	$5\cdot58$ „	$5\cdot50$ „	$5\cdot54$ „	$2\cdot756$ „	0·201	
							0·202

—From the conductivity of the solution a value for the concentration was calculated from the curve which had been already drawn. Let m be this value, and m_0 the true concentration. Then, from the standpoint of the conductivity curve, m_0 gramme-equivalents behave as though they were m gramme-equivalents. Hence, concentrations deduced from the curve were multiplied by the factor m_0/m to get the true concentrations. We get practically the same result, if, calculating k/m_0 for the solution, we increase or decrease the ordinates of the curve (by some fixed length) until it passes through the point showing the true value of k/m for the solution, and calculate concentrations from this curve. The corrections made in this way were only small ones, so that, though the method of correcting has no very firm theoretical basis, it is probably accurate enough for the purpose.

In the case of one or two of the weakest solutions made up six or seven weeks after the rest of the experiments had been performed, the discrepancy was rather large. For these solutions fresh conductivity curves were plotted (from direct experiment) which passed very near the points corresponding to the new solutions, and from these curves the "final concentrations" were calculated for these solutions.

It will be noticed that some of the values for the transport ratio are abnormally divergent from the mean. There was nothing in the course of these particular experiments to lead one to regard the results as untrustworthy. An explanation of the divergence will be suggested later. In deducing the "mean" of the results of individual experiments, however, no account was taken of this explanation; the final results therefore are purely experimental.

5. *Theoretical Considerations.*

These experiments seem to show an increase in the migration constant at extreme dilutions. If this be due to the falling-off in velocity of the hydrogen ion, we can calculate what the rise in the migration constant should be in order to account for the drop in the equivalent conductivity curve. To do this, it is necessary to make some assumption as to the cause of the smaller velocity of the hydrogen ion, and so deduce what the conductivity would have been if the velocity had remained unaltered. It has long been noticed that the phenomenon under investigation is observed only for acids and alkalis; *i.e.*, those substances which in solution form hydrogen or hydroxyl ions, the ions of the solvent. Considerations of mass action show at once that the ionisation of the water is greatly reduced by the presence of a trace of acid or alkali. The greater the concentration of the acid, the smaller will be the ionisation of the water. Assuming that the conductivity of Kohlrausch's purest water ($0\cdot87 \times 10^{-7}$) gives us approximately a measure of its ionisation in these conditions, it may be shown that for concentrations of acid or alkali greater than 10^{-7} grm.-equivalent per litre, the number of hydroxyl or hydrogen ions from the water is inversely proportional to the quantity of acid or alkali in solution.

If the diminution in the equivalent conductivity of acid solutions at extreme dilutions be caused by the water itself rather than by impurities, it is reasonable to suppose that the effect would be directly proportional to the ionisation of the water, and hence inversely proportional to the concentration of the acid, the effect becoming greater as the dilution proceeds. Now, if at points along the equivalent conductivity (k/m) curve, we add ordinates proportional to the concentration of the hydroxyl ions (*i.e.*, inversely proportional to the concentration of the acid) we obtain another curve. For one particular proportionality, and one only, this curve gradually becomes horizontal as it approaches the axis for zero concentration; that is, it neither dips nor rises. If the proportionality be greater than this critical value, the curve would rise more and more rapidly, while if a smaller proportionality be taken, the

curve would descend (in a manner similar to the actual experimental curve). It seems feasible as a working hypothesis, to regard the critical curve so obtained as giving us the k/m curve which would have indicated the conductivity, had the ionisation of the water remained unaltered.

We can now calculate the change in the migration constant which results from the diminution in conductivity—assuming that diminution to be due to the decrease in velocity of one of the ions.

Let u and v be the velocities of the anion and cation respectively, u being variable. Then at any particular concentration, the conductivity k of the acid is equal to $c(u+v)$, where c is a constant. The transport ratio μ for the anion is equal to $v/(u+v)$. Thus μk is equal to cv, and is therefore constant.

Let μ_1 and k_1 refer to the experimental curve, and μ_2 and k_2 to the theoretical one. We regard μ_2 as remaining constant. Its numerical value is obtained from experiments on solutions for which k_1 and k_2 are practically equal, for then $\mu_2 = \mu_1$. For any particular concentration we now have k_1, k_2, and μ_2; hence, μ_1, the actual migration constant, is obtained.

The process described above, of plotting the theoretical curve, was carried out with the k/m curve given in the paper already referred to (Whetham, *Roy. Soc. Proc.*, 1905, A, lxxvi., 582). Since certain impurities have a considerable effect in lowering the conductivity, it seemed feasible to take the highest curve which consistently resulted from experiment, and regard the lowness of others as being due to impurities.

Table III. gives the values then obtained, the value of μ_2 being taken as 0·186.

TABLE III.

Concentration (grm.-equiv. per litre).	k_2 (theoretical).	k_1 (experimeutal).	$u_1 = \frac{k_2}{k_1} u_2$.
0·001	367	365	0·187
0·0005	378	374·5	0·1875
0·0002	387	378·5	0·190
0·0001	389	373·5	0·1935
0·00005	390	358·5	0·2025
0·00003	390·5	339·5	0·214

The "theoretical" migration curve, plotted from the figures in the last column of Table III., is shown in the diagram (Fig. 4) as a smooth line, and the experimental values obtained (Column VIII. of table in § 4) are indicated by crosses.

§ 6. It was thought at first that these experiments gave fresh evidence in favour of the theory that the velocity of the hydrogen ion is diminished at extreme dilution, and that this diminution accounts for the drop in the k/m curve. The extent of the change observed in the migration constant seemed strongly to favour this explanation. Further examination, however, including a calculation of the effect of impurities, shows that the theory receives no support from these experiments, the whole of the rise attributed to the migration constant being readily accounted for, when we take into consideration the residual conductivity of the water. Let us then consider the problem from the standpoint of a supposed unaltered migration constant.

Let us imagine the conductivity of the distilled water to be due to the presence of some substance A B which becomes ionised in the solution. For all the substances

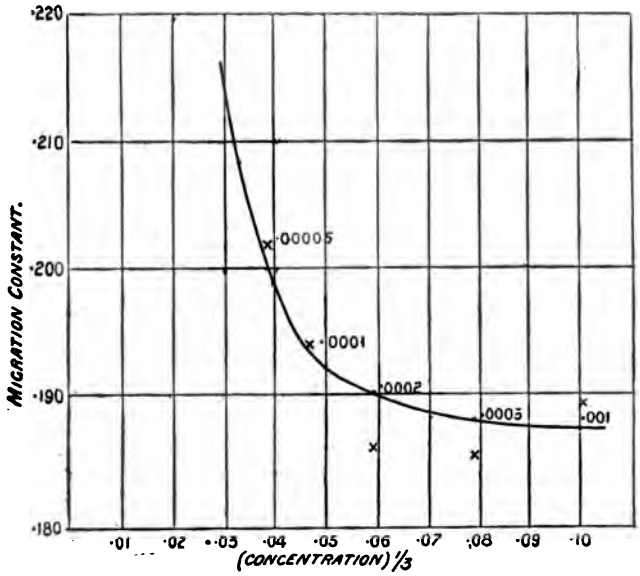

FIG. 4.

likely to constitute this impurity, hydrogen will be evolved at the cathode, and oxygen at the anode, when the solution is electrolysed. As in the electrolysis of sulphuric acid, the total quantity of solute will remain constant, and the change at the anode will be balanced exactly by that at the cathode. Consider a solution of sulphuric acid containing m grm-equivalents of the acid per cubic centimetre, and let there be, in this solution, n grm.-equivalents of the substance A B in the ionised state.

Let u be the velocity of the H ion, v of the (SO_4) ion, u' of the A ion, v' of the B ion; we have $u = 319$, $v = 73$, as deduced from the "limiting" equivalent conductivity and the migration constant of sulphuric acid.

The conductivity of the solution is $m(u+v) + n(u'+v') = K$, say. Suppose 1 grm.-equivalent of hydrogen to be liberated at the cathode. Distributing the conductivity amongst the several ions, we deduce the following changes in the anode region:—

mv/K, grm.-equiv. of SO_4 ions enters; nv'/K, grm.-equiv. of B ions enters.

mu/K, grm.-equiv. of H ions leaves; nu'/K, grm.-equiv. of A ions leaves.

Also by the action on the water of the SO_4 and B ions, liberated at the anode, 1 grm.-equivalent of H ions ar formed. Hence, in the anode region, there is a net gain

of H ions amounting to $\{mv+n(u'+v')\}/K$ grm.-equivalent.

Hence the total conductivity of the solution at the anode is increased by the amount—

$$[mv \,.\, v+nv' \,.\, v'+\{mv+n(u'+v')\}u+nu' \,.\, u']/K,$$

which is—

$$\{mv(u+v)+n(u'+v')(u+v'-u')\}/K.$$

If instead of drawing the "equivalent conductivity" curve in the usual way, we plot the specific conductivity (k) against the concentration (m), we shall find that the curve so drawn is a straight line (not passing through the origin). Hence the increase in conductivity is interpreted as being directly proportional to the increase in the concentration, and consequently (for equal quantities of hydrogen evolved at the cathode) directly proportional to the migration constant, as measured in these experiments.

For pure water and sulphuric acid, $n=0$, and the increase in the conductivity becomes $mv(u+v)/m(u+v)=v=73$.

The conductivity of the water used was about 10^{-6}. Let us regard this for the moment as being wholly due to the impurity present. For acid substances, like carbonic acid, which are only slightly ionised and have an ion in common with sulphuric acid, this number must be reduced to indicate the conductivity of A B in the acid solution, as the presence of the sulphuric acid diminishes the ionisation of carbonic acid. For the corresponding saline substances (sulphates) the suppression would be inappreciable.

1. If the impurity be free acid, we have $u=u'$, and the above expression reduces to $\{mv(u+v)+nv'(u+v')\}/K$. For the weakest solutions of sulphuric acid used, the conductivity was 18×10^{-6}, so that—

$$m(u+v)=18\times10^{-6},\quad n(u+v')=1\times10^{-6}.$$

Hence the expression for the increase in conductivity becomes—

$$(18v+v')\times10^{-3}/19\times10^{-6},$$

which is never much greater than v, since there are no anions (with the exception of the hydroxyl ion) which have a velocity appreciably greater than that of the sulphate ion. Hence the increase in conductivity at the anode, and the decrease at the cathode, is never appreciably greater than it is when no such impurity is present. With ions for which $v'<v$, the migration constant would appear to be diminished. The extreme effect for the above concentrations of sulphuric acid would be 1 part in 19 (*i.e.*, $v'=0$), reducing the measured value of the migration constant from 0·186 to 0·176. For any real ions, however, v' is never less than about 40, so that the migration constant would never appear to be less than about 0·182. For very weak acids (*e.g.*, carbonic acid) all such effects are very much reduced, as their "partial conductivity" would be much less than 10^{-6} in the presence of the sulphuric acid. We may conclude, therefore, that the presence of acid impurities in the water used as solvent cannot explain the large increase observed in the migration constant.

2. If the impurity in the water consisted of free alkali, it would be neutralised by the acid, the hydroxyl and hydrogen ions disappearing from solution. We shall find that, though the presence of traces of salt, such as potassium chloride, has no effect on the partial conductivity of the acid, yet it has a very pronounced effect upon the transport ratio as measured in the previously described experiments.

As before, we have the formula for the increase in conductivity at the anode,—

$$\{mv(u+v)+n(u'+v')(u+v'-u')\}/K.$$

Since $u(=319)$ would always be large compared with $(v'-u')$, we may get an approximate value for this expression, writiting it—

$$\{mv(u+v)+nu(u'+v')\}/K.$$

Since H and (OH) ions are excepted, v' and u' even in extreme cases, would never differ by more than 30, and hence the maximum error we introduce by taking this approximation is about 1 in 50, thereby making the transport ratio uncertain to the extent of three or four units in the third decimal place. On the theory to be suggested later, there is practically no difference between v' and u', so that the approximation is nearly exact. Putting in the values as before, we get—

$$\{18\times10^{-6}\times73+319\times10^{-6}\}/\{18\times10^{-6}+10^{-6}\}=86.$$

Hence for a solution of this strength, the migration constant appears to be increased in the ratio 86/73; *i.e.*, from 0·186 to 0·219.

This rise is greater than that actually observed. Hence, to explain the experimental results, it is only necessary to assume that about half the conductivity of the water used is due to (alkaline or) saline impurity. That such impurity should be present is more than likely; if present, its effect as described above is certain.

The physical explanation of the result of the above analysis is as follows:—If the conductivity of the water (10^{-6}) were due to the presence of sulphuric acid, the migration constant would be unaffected. When the ion A replaces the hydrogen ion, a much larger number of molecules (between three and four times as many) is required to make up the initial conductivity, 10^{-6}. Hence, in the migration, between three and four times as many B ions enter the anode region as before. Since the increase in conductivity of the solution at the anode is nearly proportional to the number of SO_4 and B ions entering the region, and since the proportion of B ions to SO_4 ions for these concentrations of sulphuric acid is quite appreciable, and the velocities of the two ions of the same order, it follows that we should expect a marked effect on the change in conductivity from which the migration constant is deduced.

§ 7. The experimental results obtained, therefore, gave no evidence in favour of the rise in the true migration constant of sulphuric acid at extreme dilutions; the whole of the apparent rise can be explained logically in a more simple way. In fact, these experiments point rather to a constant value for the transport ratio, though they would not be inconsistent with a gradual decrease in that ratio.

The above analysis also explains why the results for individual experiments sometimes varied rather widely from the mean; the supposition that a slight variation in the amount of impurity occurred, owing to the absorption of ammonia from the air, for example, being sufficient for the purpose. Also, it was noticed throughout the research, though the fact had to be left unexplained at the time, that experiments performed with different portions of the same prepared solution, generally gave results which agreed better amongst themselves than with those deduced from the use of other specimens of solution of practically the same strength. That still greater irregularity was not shown is accounted for by the fact that the various specimens of distilled water were always prepared in similar conditions, and the conductivity never varied more than 1 part in 20 from the average.

(To be continued).

CORRESPONDENCE.

THE FELLOWSHIP OF THE CHEMICAL SOCIETY.

To the Editor of the Chemical News.

SIR,—I am in entire accord with Dr. Divers that the Chemical Society is not a qualifying body. Is it too late for the Society to state this fact publicly?

It has been urged to do so for many years, and until it does, dishonest use of its Fellowship will continue.—I am, &c.,

BERTRAM BLOUNT.

7C—78, York Street, Westminster,
August 26, 1908.

NOTICES OF BOOKS.

Leather Industries Laboratory Book. By H. R. Procter, M.Sc., F.I.C., F.C.S. Second Edition. London: E. and F. N. Spon, Limited. New York: Spon and Chamberlain. 1908.

Although described as a laboratory book there is much in this work which distinguishes it from the typical practical text-book and gives it a special value as a treatise on analytical principles; the descriptions of experimental processes are clear and detailed, and in all cases the preliminary discussions of the experiments would repay very careful study before the actual laboratory work is begun; these discussions, *e.g.*, that on the chemistry of tannins, contain in most cases all that it is essential for the leather chemist to know. The book is based upon the "Text-book of Tanning," written by the author, the practical parts of which have been considerably extended for the purpose. Only thoroughly satisfactory methods which are known to fulfil the most stringent requirements as to accuracy and rapidity are given, and they are explained in detail. The applications of the microscope to leather chemistry are well treated, and a chapter is devoted to a sketch of general methods of bateriology, with references to manuals which deal with the subject more fully. Although the reader is assumed to have a knowledge of the elements of leather manufacture and chemical theory, the book is one which will be suitable for the use of the technical student from the beginning of his course, and the chemist engaged in the leather industries will also find that it contains all the information he wants for the ordinary routine work which falls to his lot, and also that it gives him much help in attacking the more difficult problems demanding originality and ingenuity with which he is frequently confronted.

The Paper Makers' Directory of all Nations. Seventeenth Edition. London: Dean and Son, Limited. 1908.

The new edition of this Directory, which has been much appreciated by all who are interested in any of the branches of the Paper trade, is somewhat larger than its predecessors, the additional matter including a short summary of the recognised customs of the trade relating to paper making in Britain, and a table of the sizes of British papers. In the lists the addresses of paper makers are classified according to various systems and are well arranged, and the Index to Commercial Prospectuses greatly simplifies reference to the many advertisements which the book contains.

MISCELLANEOUS.

Impersonation.—Consulting and analytical chemists and others are warned against receiving a tall seedy-looking man, who, on the plea of ill-health, solicits pecuniary assistance by impersonating a certain Fellow of the Institute of Chemistry in practice at Warrington.

British Association for the Advancement of Science.—The following were the Officers and Committee of Section B (Chemistry) at the Dublin Meeting of the British Association:—

President—Prof. F. S. Kipping, D.Sc., Ph.D., F.R.S.

Vice-Presidents—Sir Charles A. Cameron, M.D.; Prof. A. Smithells, B.Sc., F.R.S.; Prof. W. Noel Hartley, D.Sc., F.R.S.; Prof. Sydney Young, D.Sc., F.R.S.

Secretaries—E. F. Armstrong, D.Sc., Ph.D. (Recorder); Alex. McKenzie, M.A., D.Sc., Ph.D.; F. M. Perkin, Ph.D.; James H. Pollock, D.Sc.

Committee—Dr. W. E. Adeney; Prof. H. E. Armstrong, F.R.S.; Dr. G. H. Bailey; J. L. Baker; W. Barlow, F.R.S.; Dr. G. T. Beilby, F.R.S.; Prof. A. Brown; Dr. F. D. Chattaway, F.R.S.; H. F. Coward; H. Crompton; Sir J. Dewar, F.R.S.; Prof. A. E. Dixon; Prof. H. B. Dixon, F.R.S.; Prof. E. Divers, F.R.S.; Prof. Donnan; Prof. W. Dunstan, F.R.S.; T. Fairley; Dr. A. Findlay; Dr. L. F. Guttmann; A. D. Hall; Prof. A. G. Vernon Harcourt, F.R.S.; Dr. A. Harden; Prof. G. G. Henderson; Dr. A. Lapworth; Dr. T. M. Lowry; Dr. J. E. Mackenzie; W. MacNab; Dr. H. Marshall, F.R.S.; Dr. H. McCombie; Dr. A. Meldrum; J. H. Millar; J. O'Farelly; Prof. K. J. P. Orton; Prof. W. H. Perkin, F.R.S.; Dr. R. H. Pickard; Prof. W. J. Pope, F.R.S.; H. Ramage; Sir W. Ramsay, K.C.B., F.R.S.; R. Robinson; Prof. H. Ryan; Dr. H. J. S. Sand; Prof. A. Senier; J. L. Simonsen; Dr. A. Slator; Dr. Ida Smedley; John Smythe; Prof. J. J. Sudborough; Miss B. Thomas; Dr. J. F. Thorpe, F.R.S.; Prof. T. Turner; Dr. A. E. H. Tutton, F.R.S.; Dr. A. J. Walker; A. F. Watson; Dr. C. Weizmann; Prof. E. Werner; Dr. N. T. M. Wilsmore; Dr. M. A. Whiteley; Dr. G. Young.

The Papers brought before the Section were as follows:—

Presidential Address by Prof. F. S. Kipping, D.Sc., F.R.S.

Prof. W. H. Perkin, F.R.S.—Synthetical Experiments in the Terpene Series.

C. Weizmann—Preparation of Camphor from Oil of Turpentine.

R. Robinson—Brazilin and Haematoxylin.

Prof. W. J. Pope, F.R.S., and John Read—The Dynamic Isomerism of Oxymethylene Camphor Derivatives.

J. A. Smythe—Benzyl Sulphoxide, a possible example of tautomerism.

Dr. F. D. Chattaway, F.R.S.—(*a*) Action of Halogens upon Aromatic Hydrazines. (*b*) Some Reactions of Dichloro-urea.

Prof. F. S. Kipping, F.R.S.—Optically active Silicon Compounds.

Report of the Committee on Dynamic Isomerism.

Discussion on "Nature of Chemical Change," opened by Prof. H. E. Armstrong, F.R.S.

Dr. Ludwig Mond, Dr. H. Hirtz, Mr. D. Cowap—Note on a Volatile Compound of Cobalt with Carbon Monoxide. Communicated by Sir J. Dewar, F.R.S.

Prof. W. J. Pope, F.R.S., and William Barlow, F.R.S.—The Structure of the Open Chain Hydrocarbons.

H. Bateman—Dynamics of Chemical Action and the meaning of Valency.

Prof. H. R. Proctor—Report on Colloids.

Dr. A. Findlay—The Influence of Colloids on the Absorption of Carbon Dioxide by Water.

Dr. J. Timmermans—Densities of Liquids at Low Temperatures.

Dr. A. Meldrum—The Work of Two Irish Chemists—Bryan Higgins and William Higgins.

Julian L. Baker and H. F. E. Hulton—Action of the Enzymes of Malt on Ungerminated Cereals.

Julian L. Baker and F. E. Day—Preparation of Pure Maltose.

Discussion on "Gaseous Explosions," in Joint Meeting with Sections A and G.

Sir William Ramsay, K.C.B., F.R.S.—The Inactive Gases.

Prof. W. N. Hartley, F.R.S.—Lithium in Radio-active Minerals.

Dr. Kamerlingh Onnes—The Liquefaction of Helium. Communicated by Sir James Dewar, F.R.S.

Sir James Dewar—Anticipations and Experiments on the Liquefaction of Helium.

Prof. H. E. Armstrong, F.R.S.—(*a*) The Curriculum in Chemistry. (*b*) The Properties of Oxygen.

Dr. H. J. S. Sand—Demonstration on the Rapid Electro-analytical Separation of Metals.

W. E. Hughes and Dr. F. M. Perkin—Studies on the Electro-deposition of Metals.

Dr. J. Hübner—Mercerisation in Cotton.

Prof. T. Turner—Transparent Silver and other Thin Metallic Films.

Report of the Committee on Wave Lengths.
Discussion on "Problems of Fermentation," by Dr. A. Harden, Prof. Adrian Brown, Dr. A. Slator, Dr. E. F. Armstrong, and others.
Prof. Adrian Brown—The Selective Permeability of certain Seeds.
Discussion on Peat, by Dr. Woltereck, Capt. Sankey, Profs. Ryan, Johnson, and Lyon, Dr. Adeney, Mr. K. B. Eller, and others.
Reports of Committees—
1. Hydroaromatic Substances. 2. Aromatic Nitramines. 3. Isomorphous Benzene Derivatives.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Atacamite in Copper Ores.—Could any reader give me some information about the following:—The copper existing as atacamite in copper ores sent to Swansea is deducted from the total copper content. In the reports I have seen, the chlorine is given, but not the corresponding amount of atacamite. As I have come across many ores containing NaCl mixed with carbonates, silicates, &c., of copper, but no atacamite, I am anxious to know whether salt in any other chloride is also charged up as atacamite and the deduction made. If anyone could give me the methods of assay for chlorine employed in atacamite and other copper ores, and tell me how the whole thing is arranged, I should be obliged.—T. C. W.

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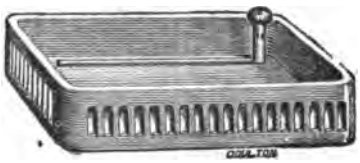

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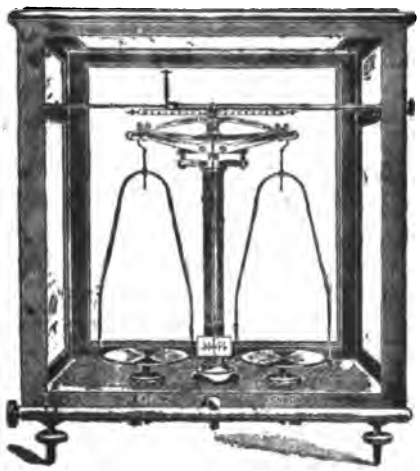

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Every candidate entering for the Matriculation Examination must pay a Fee of £2. If a candidate withdraws his name, or fails to present himself at the Examination, or fails to pass it, the Fee shall not be returned to him.

Candidates are not approved by the Examiners unless they have shown a competent knowledge in each of the following five subjects:—(1) English—Composition, Précis Writing, salient facts in English History and Geography. (2) Elementary Mathematics. (3) Latin or Elementary Mechanics, or Elementary Physics—Heat, Light, and Sound, or Elementary Chemistry, or Elementary Botany. (4) and (5) Two of the following subjects, neither of which has already been taken under Section (3). If Latin be not taken, one of the other subjects selected must be another Language from the List, either ancient or modern:—Latin, Greek, French, German, *Arabic, *Sanskrit, *Spanish, *Portuguese, *Italian, *Hebrew, and *Zoology, Ancient History, Modern History, Logic, Physical and General Geography, Geometrical and Mechanical Drawing, Mathematics (more advanced), Elementary Mechanics, Elementary Chemistry, Elementary Physics—Heat, Light, and Sound, Elementary Physics—Electricity and Magnetism, and Botany. Candidates in subjects marked with asterisk must give at least two months' notice before the commencement of the Examination.

A Pass Certificate, signed by the Principal, will be delivered to each successful candidate after the Report of the Examiners has been approved by the Senate.

Several valuable Scholarships and Exhibitions are available to students.

Intermediate Examination in Science.

The Intermediate Examination in Science will commence on the second Monday in July.

No candidate is admitted to this Examination unless he has passed, or been admitted under the Statute referred to above as exempt from, a Matriculation Examination not later than that of the preceding January.

The Fee for this Examination is £5.

Examination for Honours.

Candidates for Honours in Chemistry will be examined in Inorganic Chemistry, treated more fully than in the Pass Examination. This Examination will consist of two printed papers and a practical examination.

In the Examination for Honours, the Candidate, not being more than 22 years of age at the commencement of the Pass Examination, who most distinguishes himself will receive an Exhibition of £40 per annum for the next two years.

B.Sc. Examination.

The B.Sc. Examination will be held on the fourth Monday in October.

Candidates for this Examination are required to have passed the Intermediate Examination in Science at least one academical year previously, and to have passed the Matriculation Examination at least three years previously.

The Fee for this Examination is £5.

Examination for Honours.

For the examination for Honours in Chemistry candidates are required to show a general acquaintance with General Theoretical Chemistry, Chemistry of Carbon Compounds, Physical Chemistry, and History of Chemistry since the time of Boyle.

The candidate, being not more than 23 years of age, who most distinguishes himself in Chemistry, will receive £50 per annum for the next two years, with the style of University Scholar.

Doctor of Science.

The examination for the Degree of Doctor of Science takes place annually within the first twenty-one days of June.

No candidate is admitted to the examination for the Degree of D.Sc. until after the expiration of two Academical Years from the time of his obtaining the Degree of B.Sc. in this University.

Preliminary Scientific (M.B.) Examination.

This Examination takes place twice in each year,—once, for Pass and Honours, commencing on the second Monday in July; and once for Pass Candidates only, commencing on the third Monday in January.

The Fee for this examination is Five Pounds.

UNIVERSITY OF OXFORD.

Waynflete Professor of Chemistry—W. Odling, F.R.S.
Lees Reader in Chemistry—H. Brereton Baker, M.A., D.Sc., F.R.S.

Every Student must reside in one or other of the Colleges or Halls, or in licensed lodgings, for a period of three years. Students of Chemistry can obtain the degree of B.A. by passing preliminary examinations in Arts and in Science, and a final Honour examination in Chemistry. Chemistry may also be taken as part of the examination for a Pass degree. Graduates of other Universities and other persons suitably qualified can obtain the degree of Bachelor of Science after an approved course of study or research and two years' residence.

University Laboratory.—Demonstrators, W. W. Fisher, M.A., J. Watts, M.A., and J. E. Marsh, M.A., F.R.S., N. V. Sidgwick, M.A., A. F. Walden, M.A., B. Lambert, B.A.—The fee for students working in the Laboratory for three days in the week during the Term is £3; for students working every day, £5.

There are also laboratories which specialise in different parts of the subject:—*Physical Chemistry*, Balliol and Trinity College Laboratory: D. H. Nagel, M.A., H. B. Hartley, M.A. *Inorganic Chemistry*, Christ Church Laboratory: Dr. Baker. *Quantitative Analysis*, Magdalen College Laboratory: J. J. Manley, M.A. Jesus College Laboratory: D. L. Chapman, M.A. Queen's College Laboratory: Rev. H. B. Cronshaw, M.A.

Scholarships of about the value of £80 are obtainable at the majority of the colleges, by competitive examination in Natural Science.

More detailed information may be obtained from the Examination Statutes; the Student's Handbook to the University; and from the professors and college tutors.

UNIVERSITY OF CAMBRIDGE.

Professor of Chemistry—W. J. Pope, F.R.S.
Jacksonian Professor of Natural and Experimental Philosophy—Sir James Dewar, M.A., F.R.S.

The Student must enter at one of the Colleges or Hostels, or as a Non-collegiate Student, and keep terms for three years by residence in the University. He must pass the previous examination in Classics and Mathematics, which may be done in any term of residence or before commencing residence. He may then proceed to take a Degree in Arts, either continuing mathematical and classical study, and passing the ordinary examinations for B.A., or going out in one of the Honour Triposes.

A graduate of another University may be admitted as an "advanced" student, and obtain a degree after two years' residence in the University.

The scholarships, ranging in value from £20 to £100 a year, are chiefly given for mathematical and classical proficiency. Scholarships, or Exhibitions, are given for Natural Science at the several Colleges; the dates of the examinations vary, but are always fully advertised.

The Chemical Laboratory of the University is open daily for the use of the Students. The Demonstrators attend daily to give instruction. A list of the lectures is published annually, in June, in a special number of the *Cambridge University Reporter*, which may be had from the Cambridge Warehouse, in Paternoster Row, or through any bookseller.

Non-collegiate Students are allowed to attend certain of the College Lectures and all the Professors' Lectures, and have the same University status and privileges as the other Students. Full particulars may be obtained by forwarding a stamped directed envelope to the Assistant Registrary, Cambridge, or from the *Cambridge University Calendar*.

UNIVERSITY OF DUBLIN.

Trinity College.

Professor of Chemistry—Sydney Young, D.Sc., F.R.S.

Professor of Applied Chemistry—Emil A. Werner, F.C.S., F.I.C.

Demonstrator—W. C. Ramsden, F.C S.

Junior Demonstrators—W. R. G. Atkins, B.A., T. A. Wallace.

The general Quantitative and Research Laboratories include working accommodation for about 130 Students. The Laboratories will open on October 1st. Lectures will commence about November 1st.

The Laboratories and the Lectures ofthe Professor of Chemistry can be attended by Students who do not desire to reside in the University or proceed to its Degrees.

The full Course of General and Analytical Chemistry occupies three years, but a Student is free in his third year to devote most of his time to a special department of Pure or Technical Chemistry. Students can enter for any portion of the Course. The Lectures delivered are:—

1. *Inorganic Chemistry and Chemical Philosophy.*—Elementary, first year; Advanced Inorganic Chemistry, second year; Physical Chemistry, third year.
2. *Organic Chemistry.*—General, second year; advanced, third year.
3. *Metallurgy.*—A Course for Engineering and Technical Students.
4. *Agricultural Chemistry.*—Theoretical and Practical Courses.

The Laboratories are open every day from 10 to 5 o'clock (except Saturdays, when they close at 1 o'clock).

The Summer Course of Practical Chemistry for Medical Students begins early in April and terminates about the end of June.

A special course for Dental Students will be given.

The University of Dublin grants the Degree of Doctor of Science to graduates of Master's standing whose independent researches in any branch of Science are of sufficient merit.

By recent decrees, Prizes in Chemistry and Physics will be given in future at the October (Arts) Entrance Examination, and also at the Terminal Examinations of the Junior and Senior Freshmen Years.

Similarly, two Science Scholarships will be obtainable by Undergraduates, and tenable for five years. These Foundation Scholars receive £20 per annum, have free commons, their chambers for half the charge paid by other students, and their tutorial fees are at one-fourth the usual rates.

KING'S COLLEGE.

Senior Professor of Chemistry—J. M. Thomson, LL.D., F.R.S.

Professor—Herbert Jackson, F.C.S.

Lecturer and Demonstrators—P. H. Kirkaldy, F.C.S., H. L. Smith, B.Sc., S. W. Collins, B.Sc. and L. Hinkel.

The Academical Year consists of Three terms. The days fixed for the commencement of Terms in 1908-1909 are Sept. 30, Jan. 6, and April 28.

Students of the First Year are admitted to the Course of Theoretical and Applied Chemistry. The Course commences with a view of the conditions suitable for the production of Chemical Phenomena, after which the laws of Chemical Attraction are discussed, and the Non-metallic Elements and their principal compounds are described. The Metals and their principal compounds are next examined, care being taken to point out the applications of the Science to the Arts; and the processes of the different Manufactures and of Domestic Economy are explained and illustrated. Examinations of the Class, both *vivâ voce* and by written papers, are held at intervals during the course at the usual Lecture hour.

Second Year.—Students attend in the Laboratory twice a week, and they go through a course of Chemical Analysis, both by liquid tests and the blowpipe, analysis of ores, liquids, &c., and Elementary Volumetric Analysis.

Experimental and Analytical Chemistry in the Laboratory.—The object of this Class is to afford to Students who are desirous of acquiring a knowledge of analysis, or of prosecuting original research, an opportunity of doing so under the superintendence of the Professor and Demonstrator; Students may enter, upon payment of extra fees, at any time except during the vacation, and for a period of one, three, six, or nine months, as may best suit their convenience. The laboratory hours are from ten till four daily, except Saturday.

In addition to the Laboratory Fee, each Student defrays the expenses of his own experiments. The amount of this expense, which is comparatively trifling, is entirely under his own control.

Fees.—Chemistry: Winter Session, £5 5s.; Summer Session, £3 3s. Practical Chemistry: Winter Session, £8 8s.; Summer Session, £4 4s. Experimental and Analytical Chemistry—Five days a week: One month, £4 4s.; One Term, £10 10s.; Two Terms, £18 18s.; Three Terms, £26 5s. Three days a week: One month, £2 12s. 6d.; One Term, £6 6s.; Two Terms, £11 11s.; Three Terms, £15 15s.

For fuller details the separate Syllabuses provided for each class should be consulted.

Metallurgy.

Professor—A. K. Huntington, A.R.S.M, F.I.C., &c.

The following subjects are treated of in the Lectures: The Selection and Economic Preparation of Fuel and of Refractory Materials; the methods by which metals are obtained from their ores, and the means by which they are rendered suitable for the various requirements of the Arts.

Particular attention is paid to the study of the Nature and Properties of Metals and Alloys available for Constructive Purposes.

In the Metallurgical Laboratory, which is always open during College hours, the relation between the Chemical Composition of Metals and their Mechanical Properties may be studied by the aid of Testing Machinery.

Fee, £3 3s. for the Course.

Photography.

Lecturer—Prof. J. M. Thomson, LL.D., F.R.S.

For particulars apply to Prof. Thomson.

Evening Classes.

Classes for Evening Instruction in various subjects are held during the Winter Session.

UNIVERSITY COLLEGE.

Faculty of Science.

Professors—Sir William Ramsay, K.C.B., LL.D., F.R.S. (Inorganic and Physical Chemistry); J. Norman Collie, Ph.D., F.R.S. (Organic Chemistry).

Assistants—E. C. C. Baly, F.I.C.; S. Smiles, D.Sc.; N. T. M. Wilsmore, M.Sc.; Katharine A. Burke, B.Sc.; R. W. Gray, Ph.D.; and W. B. Tuck, B.Sc.

The Session is divided into three Terms, as follows:—First Term, from October 5 to December 18; Second Term, from January 12 to March 31; Third Term, from April 27 to July 7.

Introductory Course of Inorganic Chemistry.

Tuesday and Thursday, at 9. Practical, Thursday, 2 to 3.30, or 3 30 to 5. Fee, £10 10s.

This Course treats of the chief physical and chemical characters of the Non-metallic Elements, their preparation and characteristic tests.

Junior Course of Inorganic Chemistry.

First and Second Terms: The Class meets four times a week, on Mondays, Wednesdays, Fridays, and Saturdays, at 9, for Lectures, Examinations, and Exercises.

In this Course the physical principles bearing on Chemistry are first discussed, and the Chemistry of the Elements and their compounds is treated in considerable detail, under the headings—Elements; hydrides, halides, oxides, sulphides, &c., borides, carbides and silicides, nitrides, phosphides, &c., and alloys. Special attention is devoted to substances of commercial importance. The concluding Lectures are devoted to Physical Methods and their bearing on Chemical Problems.

Third Term: Lectures will be given on Mondays and Fridays at 9 and on Wednesdays at 11, on the chief types of Carbon Compounds.

Fees: Course, £7 7s.; First or Second Terms, £4 4s.

A Practical Class will meet throughout the Session.

Course of Physical Chemistry.

October to February: Tuesday and Thursday at 9. February to June: Monday and Saturday at 9.

The Principles of Chemistry will be specially considered, comprising the Atomic Theory, Chemical Classification, the Periodic Law, Thermal Chemistry, Dissociation, the Application of Physical Methods to the Solution of Chemical Problems, and the influence of Mass and Temperature on the progress of Reactions. Special attention is paid to the most recent Chemical Theory researches.

Fees:—Courses, £5 5s.; Term, £2 2s.; for a second course, £1 1s.

Senior Course of Inorganic Chemistry.

This Course will begin about the middle of February; Tuesday and Thursday at 9. Fee, £3 3s.

Organic Chemistry.

Mon. at 12, Wed. and Fri. at 9, during the session.

This Course is suitable for Students who intend to study Chemistry from a scientific standpoint. No previous knowledge of Organic Chemistry is expected of those attending the Class, which should, however, be taken concurrently with the Course of Physical Chemistry, and with Practical Organic Chemistry in the Laboratory.

Fee:—For the Course, £6 6s.; for a Term, £2 12s. 6d.; for a Second Course, £3 3s.

An Advanced Course will be held twice a week throughout the Session for those engaged in prosecuting research in Organic Chemistry. Fee, £5 5s.; Term, £2 2s.

Practical Classes.

Practical Classes in Inorganic and Organic Chemistry are conducted by the Assistants.

Laboratories of General and of Organic Chemistry.

The Laboratories are open daily from 9 a.m. to 5 p.m., Saturdays excepted, from October until the middle of July, with a short recess at Christmas and at Easter.

Fees: for the Session, £26 5s.; six months, £18 18s.; three months, £10 10s.; one month, £4 4s.

Three specified days a week:—for the Session, £15 15s.; six months, £11 11s.; three months, £6 6s.; one month, £2 12s. 6d., exclusive of expense of materials. Students may enter at any period of the Session.

When accompanied by, or preceded by, attendance on the Lectures on Inorganic and Organic Chemistry, the Laboratory Courses-qualify Students in the application of Chemistry to Manufactures, Metallurgy, Medicine, or Agriculture, &c.

There is also a Chemical Library containing the chief Journals and Standard Works on Chemistry.

Certificates of Honour are granted to competent Students on the work done during the Session. Several valuable Scholarships are available to students.

IMPERIAL COLLEGE OF SCIENCE AND TECHNOLOGY.

Professor of Chemistry—W. A. Tilden, D.Sc., F.R.S.

Assistant Professors—M. O. Forster, Ph.D., D.Sc., F.R.S.; G. T. Morgan, D.Sc., A.R.C.S.; J. C. Philip, M.A., Ph D, B.Sc.

The Imperial College of Science and Technology, incorporated under the Royal Charter of July 8, 1907, is an institution or group of associated colleges with its principal seat at South Kensington.

The purposes of the Imperial College are to give the highest specialised instruction, and to provide the fullest equipment for the most advanced training and research in various branches of science, especially in its application to industry; and to do all and any of such things as the Governing Body consider conducive or incidental thereto, having regard to the provision for those purposes which already exists elsewhere.

For these purposes, the Governing Body, subject to the provisions of the Charter, are to carry on the work of the Royal College of Science, and the Royal School of Mines, and may establish Colleges and other Institutions or Departments of Instruction. Any Institution or Department so established, and, subject to the conditions of the Charter, the Central Technical College of the City and Guilds of London Institute, are to be integral parts of the Imperial College; and the Central Technical College is to be called and known in future as the City and Guilds College.

Subject to agreement with the authorities of any College or other Institution, the Governing Body may by resolution recognise that College or Institution or any Department thereof, as being in association with the Imperial College for all or any of the purposes of the Charter, but no such resolution is to be valid or operative until allowed by the King in Council.

The Charter further provides that the Imperial College shall be established in the first instance as a School of the University of London. Students of the Imperial College who have matriculated at the University of London may therefore proceed to the Science degree of the University as "Internal Students."

The Session 1908-9 will see certain modifications of the work as carried on hitherto by the constituent Colleges, and while intending students can rely generally on the carrying out of the scheme as outlined, it must be understood that it may be subject to modification in detail.

Attention is particularly directed to the conditions of admission and to the extended facilities for Research Work.

The ordinary courses of instruction are planned so as to extend over four years instead of three years, as heretofore, in the several constituent Colleges. They are generally similar for all students during the first year, and to a less extent during the second year, after which they are specialised according to the particular course which the student is taking.

The following Diplomas have been awarded in the past to Students of the several constituent Colleges:—

The Associateship of the Royal College of Science (A.R.C.S.) in one or more of the following divisions:—Mechanics, Physics, Chemistry, Biology, Geology.

The Associateship of the Royal School of Mines (A.R.S.M) in one or more of the following divisions:—Metallurgy. Mining.

The Associateship of the City and Guilds Institute (A.C.G.I.) in one or more of the following divisions:—Chemistry, Engineering (Civil and Mechanical), Engineering (Electrical).

The instruction in Chemical Science embraces:—

1. Courses of lectures on the laws and general principles of Chemistry, including the special study of compounds of carbon, usually called "Organic Chemistry"; on the theory and methods of Chemical Analysis; and on Physical Chemistry.

2. A systematic Laboratory course for the practice of Qualitative and Quantitative Analysis.

An examination will be held at the end of each course of instruction, and all candidates for a Diploma in Chemistry will be required to qualify in each successive stage.

A Laboratory course is provided for advanced students desirous of acquiring a knowledge of the methods employed in research and for the prosecution of Chemical Investigations.

Full details can be obtained from the College Calendar, published by Eyre and Spottiswoode (or through any bookseller), price 6d.

THE SCHOOL OF PHARMACY OF THE PHARMACEUTICAL SOCIETY OF GREAT BRITAIN.

The Sixty-sixth Session will commence on Sept. 30, 1908.

Professors—Chemistry, Arthur W. Crossley, D.Sc., Ph.D., F.I.C., F.R.S., (Dean); Pharmaceutics, Henry G. Greenish, F.I.C., F.L.S.

A Course of Lectures on Physical, Inorganic, and Elementary Organic Chemistry commences in October and terminates at the end of June. An Advanced Course of Lectures begins in October and extends to the end of March. These Lectures are adapted to the requirements of Pharmaceutical and Medical Students, and also of those who are proceeding to degrees at the University of London, or who are preparing for the examinations of the Institute of Chemistry.

Entries may be made for single classes. Certificates of attendance at the two Courses of Lectures on Chemistry and at the Chemical Laboratories are accepted as evidence of chemical training by the Institute of Chemistry in connection with the Examinations for the Associateship, and also by the Conjoint Board of the Royal Colleges of Physicians and Surgeons, as well as by other examining bodies. Certificates of attendance at the Course of Pharmacy is also accepted by the Conjoint Board.

Prospectuses and further information may be obtained from the Dean of the School, 17, Bloomsbury Square, London, W.C.

UNIVERSITY COLLEGE OF WALES, ABERYSTWYTH.

UNIVERSITY OF WALES.

Director of the Edward Davies Chemical Laboratories and Professor—J. J. Sudborough, D.Sc. (Lond.), Ph.D. (Heidelberg), F.I.C.

Assistant Lecturers and Demonstrators—A. Brooke, Ph.D. (Strass.), and T. C. James, M.A. (Cantab.), B.Sc.

Lecturer in Agricultural Chemistry—J. Jones Griffith, B.Sc. (Wales).

The College is open to men and women students above the age of sixteen years. The Session commences on Sept. 29th, on which day all Students will be expected to meet the Professors in the Examination Hall of the College.

Lecture Courses.—(1) Intermediate Science Course; four lectures weekly throughout the Session. (2 and 3) B.Sc. Courses; A, three lectures weekly on Organic Chemistry; B, three lectures weekly on General and Physical Chemistry. (Courses A and B will generally be given in alternate Sessions; for 1908-1909, Course B). (4 and 5) Courses in Agricultural Chemistry. For students in their first year, 3 lectures, and for those in their 2nd year, 2 lectures weekly during the Michaelmas and Lent terms.

Laboratory Courses.—The Laboratories are open daily from 9 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays. Regular Courses of practical work, suitable for the B.Sc. degree of the Universities of London and Wales, or for the Associateship of the Institute of Chemistry, can be followed. Facilities are given for Students wishing to undertake research work. Special Courses will be arranged for those who intend to follow Medicine or Pharmacy, or any one particular branch of Applied Chemistry, always provided that such Students possess the requisite knowledge of Theoretical Chemistry. The hours will be arranged, as far as possible, to suit the requirements of the individual Student.

The College is recognised by the University of Edinburgh and the Royal University of Ireland, and by the Colleges of Physicians and Surgeons of England, Scotland, and Ireland as an institution at which the instruction necessary for their respective Diplomas in Medicine, in Chemistry, Physics, and Biology may be given. One year for graduation in Medicine and two years for graduation in Science may be spent at Aberystwyth.

Fees.—The Fee for the whole Session, if paid in advance, is £10; if paid by Single Terms, for the first term of attendance in each Session, £4; for the second term, £3 10s.; for the third term, £3. These composition fees enable the Student to attend any or all the Classes of the College, with the exception that a small extra fee is charged for Laboratory Instruction. Thus, for Practical Chemistry, the additional fee is, for six hours' work per week, 12s. 6d. per term, and for twelve hours, 25s. per term. The fees for those who desire to spend several days weekly in the laboratory may be learned on application to the Registrar. Fee for a single Lecture Course £1 per term.

Scholarships and Exhibitions varying in value from £10 to £40 per annum will be offered for competition at examinations which commence on September 15, and exhibitions are awarded at the end of the Session on the results of the class examinations.

Intending Students requiring further information are recommended to write to the Registrar for a copy either of the General Prospectus or of one of the Special Prospectuses issued for the Agricultural and Normal Departments.

UNIVERSITY COLLEGE OF NORTH WALES, BANGOR.

A CONSTITUENT COLLEGE OF THE UNIVERSITY OF WALES.

Chemistry.—Professor, K. J. P. Orton, M.A., Ph.D., F.I.C. Assistant Lecturer and Demonstrator, Alice E. Smith, B.Sc. Assistant Lecturer in Agricultural Chemistry, R. Gaunt, Ph.D., B.Sc. Junior Demonstrator, J. O. Hughes, B.Sc.

Physics.—Professor, E. Taylor Jones, D.Sc. Assistant Lecturers and Demonstrators, A. H. Verguson, B.Sc., and W. E. Williams, B.Sc.

The Session opens September 29th, 1909. All regular classes are open to men and women students above the age of 16 years. The following Courses of Lectures will be given.

Intermediate Course.—Inorganic Chemistry and Elementary Physical Chemistry. Fee for the Session, £3 13s. 6d.

B.Sc. Course.—Organic Chemistry. Fee for the Session, £3 3s. Advanced Lectures on Organic Chemistry, £1 1s.

Agricultural Chemistry.—Fee, £2 2s.

Laboratory Courses.—The laboratory is open on five days of the week from 10 a.m. to 5 p.m. for instruction in Chemical operations and in the Application of Chemistry to Medicine and the Industrial Arts. Fees: six hours per week, £1 1s. per Term; twelve hours, £2 2s.; eighteen hours, £3 3s. Composition Fee for all Laboratory Classes of the Intermediate Science Course taken in one year, £4 4s.

The Chemistry, Botany, Zoology, and Physics Courses are recognised for Medical graduation in the Universities of Edinburgh and Glasgow, and by the Conjoint Boards of the Royal Colleges of Surgeons and Physicians, and students can make one *Annus medicus* at the college. Students are prepared for the First Examination of the Universities mentioned, and of Conjoint Board of the

Royal Colleges of Surgeons and Physicians. The Science Courses are recognised for part of the science degree course of the University of Edinburgh.

UNIVERSITY COLLEGE OF SOUTH WALES AND MONMOUTHSHIRE, CARDIFF.

Professor—C. M. Thompson, M.A., D.Sc., F.C.S.
Assistant Professor—E. P. Perman, D.Sc., F.C.S.
Demonstrator—R D. Abell, D Sc.
Lecturer in Metallurgy—A. A. Read, F.I.C., F C.S.

The Session commences October 6th, and terminates on June 15th, and is divided into three terms.

The Junior Course (delivered during the Michaelmas term only) consists of about 50 lectures, and will cover the subjects prescribed for the Matriculation examinations of the University of Wales and the University of London. Fee, £2 2s.

The Intermediate Course consists of about 80 lectures held during the Lent and Summer terms in continuation of the Junior Course; together with laboratory practice it forms the qualifying course for the Intermediate Examination of the University of Wales, and will cover the subjects required for the Intermediate Examination in Science (Part I.) of the University of London. Fee, £4 4s.

The Senior Course consists of about 80 lectures on Inorganic Chemistry; Fee, £3 3s.

The following lectures on Metallurgy will be given by Mr. Read:—10 lectures on Fuel; Fee, 10s. 6d. 20 lectures on General Metallurgy; Fee, £1 1s. 30 lectures on the Manufacture of Iron and Steel; Fee, £1 1s. A practical course on Iron and Steel Analysis will also be held, and practical instruction in Dry Assaying will be given in the Metallurgical Laboratory, which is fitted with the necessary furnaces and apparatus.

In the laboratory each student works independently, so that the course of study may be adapted to the requirements of the individual. Hours, 9 to 1 and 2 to 5; Saturday, 9 to 1. Fees—Six hours per week, £2 2s. per term; twelve hours, £3 3s. per term; eighteen or more hours, £4 4s. per term.

Registered medical students can prepare for the Intermediate M.B. Examination of the University of London, and spend three out of their five years of medical study in Cardiff. Medical students wishing to graduate at a Scottish University, or preparing for a Conjoint Board Surgical and Medical Diploma, or for the Diploma of the Society of Apothecaries, can spend two years in Cardiff. For further information see the prospectus of the Faculty of Medicine, which may be obtained from the Registrar.

The College is recognised as an institution at which two years of the course for the degree of Bachelor of Science of the University of Edinburgh may be spent.

Students by making a payment of £10 at the commencement of each session may compound for all lecture fees for the whole session. Laboratory fees are not included in the composition fee, but Students preparing for the Science Examinations of the University of Wales and of the University of London may, by making a payment of £13 13s. at the commencement of each Session, compound for both Lecture and Laboratory Fees during the Session.

At the entrance examination in September, and the annual examination in June, several scholarships and exhibitions are awarded. Great importance is attached to special excellence in one subject.

The College Prospectus, and also further information as to scholarships, may be obtained from the Registrar.

A Hall of Residence for Women Students is attached to the College.

UNIVERSITY COLLEGE, BRISTOL.

Professor of Chemistry—Francis Francis, D.Sc., Ph.D., F.I.C.
Lecturer (Physical Chemistry)—James W. McBain, M.A.
Lecturer—Oliver C. M. Davis, B.Sc., F.I.C.
Lecture Assistant—J. H. Sturgess.

The session 1907-1908 will begin on October 1. Lectures and classes are held every day and evening throughout the Session. In the Chemical Department lectures and classes are given in all branches of theoretical chemistry, and instruction in practical chemistry is given daily in the chemical laboratory. A Laboratory has recently been fitted with a complete equipment for all ordinary measurements in Physical Chemistry. The department of experimental physics includes various courses of lectures arranged progressively, and practical instruction is given in the physical and electrical laboratories. The Department of Engineering and the Constructive Professions is designed to afford a thorough scientific education to students intending to become engineers, or to enter any of the allied professions, and to supplement the ordinary professional training by systematic technical teaching. This department includes courses specially arranged for students intending to become civil, mechanical, electrical, or mining engineers, surveyors, or architects. Those who attend the mechanical engineering course enter engineering works during the six summer months, and, in accordance with this scheme, various manufacturing engineers in the neighbourhood have consented to receive students of the College into their offices and workshops as articled pupils at reduced terms. Medical education is provided by the Faculty of Medicine of the College. Several Scholarships are tenable at the College. Full information may be obtained from the Registrar.

Day Lectures.

Inorganic Chemistry.

The Courses treat of the principles of Chemistry, and of the Chemistry of the Non-Metals and Metals.

Introductory Course.—Three Lectures a week will be given during the First and Second Terms, and at least one Lecture a week during the Third Term. Fee, £3 13s. 6d.

General Course.—Three Lectures a week will be given throughout the Session. Fee, £3 13s. 6d.

Systematic Physical Chemistry (a course extending over two Sessions).—Two Lectures a week will be delivered throughout the Session. Fee, £3 13s. 6d.

Electro-chemistry.—One Lecture a week will be given during the Third Term. Fee, £1 1s.

Advanced Course.—Two Lectures a week will be given throughout the Session. Fee, £3 13s. 6d.

Organic Chemistry.

General Course—Three Lectures a week will be given throughout the Session. Fee, £3 13s. 6d. An advanced course of lectures will also be given one day a week during the session. Fee, £2 2s.

Colloquium.

A weekly Colloquium will be held by members of the Staff to discuss recent advances in the various departments of Chemistry. Students and others interested are invited to attend.

Practical Chemistry.—Laboratory Instruction.

The Chemical and Metallurgical Laboratories will be open daily from 9 a.m. to 5 p.m., except on Saturdays, when they are open to senior students only. Instruction will be given in the Laboratory in all branches of Inorganic, Organic, and Physical Chemistry. Special facilities will be afforded to those who desire to carry out research or to study Practical Chemistry as applied to the different processes employed in the Arts and Manufactures. Fees in Guineas—

Days per Week ..	Five.	Four.	Three.	Two.	One.
Per Session	15	12½	10	7½	5
,, Two Terms ..	11	9	7½	5½	3½
,, One Term ..	7	6	4½	3½	2½

Students may arrange to divide their days of laboratory work into half-days. Special facilities are afforded for Research and Post-graduate work.

Chemical Scholarship.—Among others, a Chemical and a Metallurgical Scholarship each of £25 are offered for competition.

Evening Lectures.

A course of Lectures dealing with the principles and theories of modern Chemistry will be delivered during the First and Second Terms. Fee, 10s. 6d.

Practical Chemistry — Laboratory Instruction. — The Laboratory will be open one evening a week from 6 till 9. Instruction will be given in Qualitative and Quantitative Analysis, and in the Preparation of Chemical Products Fees:—Two Terms, 21s; One Term, 12s. 6d.

University College, Bristol, has been approved by the Council of the Institute of Chemistry as a College at which all the subjects required for the admission of Associates to the Institute are taught.

The Calendar of the College, price 1s. (post-free, 1s. 4d.), containing detailed information of the various Courses, may be obtained on application to the Registrar.

MERCHANT VENTURERS' TECHNICAL COLLEGE, BRISTOL.

Chemistry.

Professor—J. Wertheimer, B.Sc., B.A., F.I.C., F.C.S.
Assistant Professor—Frederick W. Rixon, M.Sc., Ph.D.
Lecturers—H. A. M. Borland, A.R.C.S.; E. E. Elt, B.Sc
Demonstrators—E. H. T. Parker; D. A. Baker.
Assistants in Chemical Laboratory—W. F. Phillips and C. Cox.

The Laboratories of this Department are open during the Term on every week-day as a School of Practical Chemistry and Analysis to Students of either sex. Each Student works independently, and receives individual instruction and help.

The training in this Department is intended to fit Students to become Professional Chemists, to prepare them for Medical or Pharmaceutical examinations in Chemistry, to instruct them in the branches of Applied Chemistry required in Manufactures and Agriculture.

Special facilities will be given to Students wishing to undertake research work approved by the Professor; and the Courses provide for the Examinations in Chemistry for the B.Sc., M.B., and D.Sc. degrees of the University of London.

The three years' course at this College in the subjects of Theoretical and Practical Chemistry, Theoretical and Practical Physics, and Elementary Mathematics, is accepted by the Council of the Institute of Chemistry as satisfactory evidence of the training of candidates for the Associateship of the Institute.

The Laboratories are open every week-day from 9.30 till 12.30, and from 2 to 5 p.m., except Saturday, when they close at 12.30.

The Annual Fee of £10 10s. secures (in addition to the use of the Laboratory) permission to attend the Lectures in Physics and Mathematics as well as Chemistry, and to use the Physical Laboratory and Engineering Workshop.

The College Day Classes re-open on Sept. 15, and the Evening Classes commence on Monday, September 28.

Further detailed information may be obtained from the College Calendar, price 6d., or the Prospectus for Day or Evening Classes (free).

UNIVERSITY OF BIRMINGHAM.

Professor—Percy F. Frankland, Ph.D., M.Sc., LL.D., F.R.S., P.I.C.
Assistant Lecturers and Demonstrators—Alex. Findlay, M.A., D.Sc., Ph.D.; Hamilton McCombie, M.A., Ph.D., B.Sc., A.R.C.S., A.I.C.; C. K. Tinkler, D.Sc., and T. J. Murray, Ph.D., M.Sc.

The Session will be opened on October 1st, 1908.

Lecture Courses.

First Year.—A. This part of the course is arranged (1) to give a full exposition of the general principles of Chemical Science, (2) for the systematic study of the properties of the more important elements and their compounds, and (3) to indicate the chief applications of Chemistry in the Arts and Manufactures. Four hours weekly during the Winter and Spring terms. Mondays to Thursdays inclusive, at 9.30 to 10.30 a.m. Fee, £5 5s. B. This part of the course includes an introduction to the study of Organic Chemistry, with a description of the properties, relations, and methods of preparation of the more important groups of Carbon Compounds. Three hours weekly during the Summer term. Mondays, Wednesdays, and Fridays, at 9.30 to 10.30 a.m. Fee, £1 11s. 6d.

Second Year.—Advanced Organic Chemistry.—This course extends over two years, and is divided into two parts:—(*a*) Carbon Compounds of the Fatty Series; (*b*) Aromatic and other Cyclic Compounds. Only one of these parts will be taken in each year. The class meets twice weekly by arrangement during the Winter and Spring terms. Fee, £2 2s. *General and Physical Chemistry.*—This course will deal in outline with the more recent developments of Theoretical Chemistry. The class meets once weekly by arrangement during the three terms. Fee, £1 11s. 6d.

Third Year.—A further Course in Advanced Organic Chemistry will deal with one of the above parts of the Course. The class meets two hours weekly by arrangement during the Winter and Spring terms. Fee, £2 2s. A further Course on General and Physical Chemistry, in which special subjects attracting attention at the time receive treatment. Fee, £1 11s. 6d.

Practical Chemistry.

The instruction in Practical Chemistry extends over three years. The Laboratory will be open daily from 9.30 to 5, except Saturdays, when it closes at 1 p.m. Fees—

	All day.	Three hours per day.	Three hours per day; five days a week.	Three hours per day; three days a week.
	Guineas.	Guineas.	Guineas.	Guineas.
One Term ..	7	4½	4	2½
Two Terms .	13	8½	7½	5
Three Terms	18	12	11	6½

A Course of short demonstrations and exercises is given by the Professor or one of his Assistants once a week. All first-year Students are required to attend, unless exempted for special reasons by the Professor. No Fee.

Special facilities are given to Advanced Students for the prosecution of original research.

Metallurgy.

There is a separate University department for Metallurgical students in which provision is made for instruction in assaying, &c.

Scholarships.

Priestley Scholarships.—Three Open Scholarships in Chemistry of the value of about £96 each are awarded annually in June.

Ascough Scholarship.—One Open Scholarship of the value of about £30 is awarded annually in July.

Bowen Scholarship.—One Open Scholarship in Metallurgy, value about £96, is awarded annually in June.

For particulars apply to the Registrar.

Excursions.

During previous Sessions permission has been obtained to visit some of the great factories in or near Birmingham, in which chemical and metallurgical industries are carried on. Students have thus had most valuable opportunities of gaining a practical acquaintance with some branches of Applied Science. The privilege thus courteously granted by several manufacturers will, it is hoped, be enjoyed in every future Session. The excursions will be conducted by the Professor or Lecturers.

CITY OF BRADFORD TECHNICAL COLLEGE.

Principal—Prof. W. M. Gardner, M.Sc.

Department of Chemistry and Dyeing.

Professor of Chemistry and Dyeing—W. M. Gardner, M.Sc.
Assistant Professor of Chemistry—B. North, A.R.C.Sc. (Lond).
Lecturers in Chemistry—L. L. Lloyd, Ph.D.; H. H. Hodgson, M.A., B.Sc., Ph.D.; S. F. Stell, F.C.S.

Demonstrators in Chemistry—A. E. Findley, B.Sc.; S. M. Sutcliffe, F.C.S.

Lecturer in Physics—J. A. Tomkins, A.R.C.Sc. (Lond.).

Demonstrator in Physics—E. B. Walker.

Lecturer in Dyeing—A. B. Knaggs, F.C.S.

Demonstrator in Dyeing—T. Brooke.

Lecturer in Gas Manufacture—W. Cranfield.

Lecturer in Botany, Biology, and Pharmacy—W. West, F.L.S., Ex. Pres. Yorkshire Naturalists' Union.

The following courses of instruction are provided:—

I. *General Chemistry Course*, extending over four years, including Lectures in Inorganic, Organic, and Technological Chemistry, Principles of Analysis, Technical Analysis, Electro-chemistry, Physical Chemistry, Crystallography, Fuels, Lighting and Ventilation, Physics, Mathematics, Mechanics, with Laboratory work in Chemistry, Physics, Bacteriology, and Microscopy.

II. *Chemistry and Dyeing*, extending over four years. Includes most of the above subjects, along with Lectures and practical work in Dyeing, Colour matching, &c.

III. *Chemical Engineering*. Three years' course, preparing Students for positions in Chemical Works, Sewage Works, &c.

IV. *Sanitary Science*. One year's Course, recognised by the Sanitary Institute as preparing for their certificate examination. Subjects: Chemistry, Physics, Sanitary Engineering, Sanitary Law, Building Construction, Drawing, Physiology, and Bacteriology.

V. *Dyeing*. Extending over one or two years.

VI. *Textile and Dyeing*. Arranged for those Students who desire to study the two subjects simultaneously.

VII. *First Professional Examination, Conjoint Medical Board (M.R.C.S., L.R.C.P.), London*.—Attendance at the College and College Certificates in Chemistry, Physics, and Biology are recognised by the Conjoint Board for Medical Studies as a qualifying curriculum.

VIII. *General Pharmaceutical Course*. Prepares for the Minor and Major Pharmaceutical Examinations. Each extends over two years on four half-days per week, and includes Chemistry and Physics, Botany, Biology, Materia Medica, Pharmacy, and Dispensing.

ROYAL AGRICULTURAL COLLEGE, CIRENCESTER.

Chemical Department.

Professor—Prof. E. Kinch, F.C.S., F.I.C.

Assistant—W. James.

Systematic courses of Lectures are given on the various branches of Chemistry in its relation to Agriculture, illustrated by experiments, and by the collections in the College Museum. They comprise the laws of Chemical Combination and the general Chemistry of mineral bodies, and of the more frequently occurring bodies of organic origin, with the relationships of their leading groups; and, finally, the applications to practical operations of the Chemistry of the atmosphere, of soils and manures, of vegetation, of stock feeding, and of the processes and products of the dairy.

In the Laboratory practical instruction is given in the construction and use of apparatus and in Chemical manipulation and analysis, both qualitative and quantitative. After studying the simple operations and the properties of the commonly occurring substances, the Students are taught to analyse a series of compounds, and apply the knowledge thus obtained to the analysis of manures, soils, waters, feeding stuffs, dairy products, and other substances met with in the ordinary course of Agricultural practice. Chemico-agricultural researches are undertaken by the senior Students under the direction of the Professor and his Assistants.

THE UNIVERSITY OF LEEDS.

Professor of Chemistry—Arthur Smithells, B.Sc., F.R.S.

Professor of Organic Chemistry—Julius B. Cohen, Ph.D, B.Sc.

Lecturer on Physical Chemistry—H. M. Dawson, D.Sc, Ph.D.

Assistant Lecturers and Demonstrators—W. H. Perkins, B.Sc.; W. Lowson, B.Sc., F.I.C.

Demonstrators—H. Calam, M.Sc.; A. T. King, B.Sc; J. Marshall, B.Sc.

The Session begins October 1, 1908.

Lecture Courses.

1. General Course of Chemistry.—Monday, Wednesday, and Friday, at 11.30 a.m. Fee for the Course, £4 4s.

1a.—Supplementary Course of Inorganic and Organic Chemistry. Fee, £3 13s. 6d.

2. Advanced Inorganic Chemistry.—Metals. Monday, Wednesday, and Friday, at 9.30 a.m. Fee, £3 13s. 6d.

3. Advanced Inorganic Chemistry.—Non-metals. Tuesday, Thursday, and Saturday, at 9.30 a.m. Fee, £3 13s. 6d.

4. Organic Chemistry.—Tuesday, Thursday, and Saturday at 12 noon Fee £3 13s. 6d.

5. Honours Courses—(*a*) Organic Chemistry: Monday, Wednesday, and Friday at 12 noon in the First and Second Terms; fee, £2 12s. 6d. (*b*) History of Chemistry: Monday, Wednesday, and Friday at 9.30 a.m. in the First Term; fee, £1 11s. 6d. (*c*) Physical Chemistry: Monday, Wednesday, and Friday at 9.30 a.m. in the Second and Third Terms; fee, £2 12s. 6d. (*d*) Electro-chemistry: One hour weekly; 31s. 6d.

6. Chemistry of Food and Drugs.—Special class for Students taking Final Examination of the Institute of Chemistry in Branch E (Food and Drugs). £2 2s.

Laboratory Courses.

The University Laboratory will be open daily from 9 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it will close at 1 p.m.

Fees for the Session.—Students working six days per week, £21; five, £18 18s.; four, £16 16s.; three, £13 13s.

Practical Course in Sanitary Chemistry.—Tuesdays and Thursdays from 2 to 5 from April to June. Fee, £5 5s.

Tinctorial Chemistry and Dyeing Department.

Professor—Arthur G. Green, M.Sc., F.I.C.

Lecturer and Research Chemist—A. G. Perkin, F.R.S., F.I.C.

Assistant Lecturer—Ian Q. Orchardson, B.Sc.

Demonstrator—G. H. Frank, B Sc.

The Courses extend over periods of three or four years, and are intended for those who wish to obtain a full scientific and practical education in the art of dyeing, colour manufacture, &c. It is suitable for those who purpose in the future to take any part in the direction of the operations of dyeing or printing of textile fabrics, *e.g.*, the sons of manufacturers, calico printers, managers, master dyers, &c., or in the manufacture of coal-tar products and dyestuffs.

Leather Industries Department.

Professor—H. R. Procter, M.Sc., F.I.C.

Assistant Lecturer and Demonstrator—H. G. Bennett, M.Sc.

Demonstrator—Harold Brumwell.

Research Assistant—D. J. Law, B.Sc., A.I.C.

The full Courses, which extend over a period of either two or three years, are suitable to all who intend to become Technical Chemists in the Leather Industry, or managers of important works, and are recommended to sons of tanners. The Courses include instruction in chemistry, a modern language, leather manufacture, and practical work in the Leather Industries Laboratory and Dye-house.

Agricultural Department.

Professor—R. S. Seton, B.Sc.

Lecturer in Agricultural Chemistry—C. Crowther, M.A., Ph.D.

The full Course occupies three years, and includes instruction in chemistry, physics, mathematics, geology, botany, forestry, engineering and surveying, and the principles of agriculture, as well as practical work in the various laboratories and out door agriculture at the University Farm.

Fuel and Metallurgical Department.

Professor—William A. Bone. D Sc., Ph.D, F R.S.

The Courses extend over two, three, or four years, and are suitable for those who are preparing for posts either as Gas Engineers or in Fuel and Metallurgical industries.

The Courses in Gas Engineering and the Technology of Fuel will chiefly deal with the manufacture and distribution of coal-gas and gas-lighting problems, by product coking processes, and the production and application of gaseous fuels for heating and power purposes.

The Metallurgical Courses, besides dealing with general processes for the concentration and extraction of ores, will be chiefly directed to problems underlying blast furnace and open-hearth steel practice, and to the microstructure, physical properties, and heat treatment of steel and other industrial alloys.

Research Students are admitted to the University Laboratories on reduced terms.

Several valuable Fellowships and Scholarships are at the disposal of the University, including a Fellowship of £100 offered by the Institution of Gas Engineers, and the Salt, Akroyd, Brown, Baines, Emsley, Craven, Leeds City Council, and Clothworkers' Scholarships, and one of the 1851 Exhibition Scholarships. The North, East, and West Ridings County Council's Scholarships are tenable at the University of Leeds.

UNIVERSITY OF LIVERPOOL.

Professor of Chemistry—J. Campbell Brown, D.Sc.

Professor of Physical Chemistry—F. G. Donnan, M.A. Ph.D.

Lecturer on Organic Chemistry—A. W. Titherley, D.Sc., Ph.D.

Lecturer on Metallurgy — Guy D. Bengough, M.A., A.R.S.M.

Demonstrators and Assistant Lecturers — A. T. de Mouilpied, M.Sc., Ph.D.; H. Bassett, B.Sc., Ph.D., D.es.Sc.; F. J. Brislee, D.Sc.; A. Rule, M.Sc., Ph.D.; E. Garratt, M.Sc.; and T. E. Gardner, B.A., B.Sc.

Assistant—H. H. Froysell.

The Session commences October 5th.

Entrance Scholarship Examination takes place early in May each year.

The Classes meet the requirements of candidates for the Ordinary B.Sc. Degree, for Chemistry Honours, or for the M.Sc. or D.Sc. Degree in the University of Liverpool; for Degrees in Medicine of Liverpool; for the Pharmaceutical, Veterinary, Dental, and Public Health Diplomas; and for those studying Chemistry as a preparation for professional, technical, or commercial life. The Classes qualify for the Fellowship of the Institute of Chemistry and other Examination Boards.

Lecture Courses.

A. General Course on the principal non-metallic elements and the most important metals, the principles of Chemical Philosophy, and an introductory sketch of Organic Chemistry. Three Terms. Fee, £4.

Engineer's Course of Lectures with Practical Class. Two Terms. Fee, including Practical class, £4 10s.

Pharmacy Courses: Junior, £3; Senior, £3.

Course B.—Inorganic Chemistry. Fee, £3.

Course C.—Inorganic Chemistry (Honours Course). Fee, £2 10s.

Course D.—Organic Chemistry. Fee, £3.

Course E.—Organic (Honours). Fee, £4.

Course F.—Physical Chemistry. Fee, £3.

Course G.—Honours Physical Chemistry. Fee, £4.

Course H.—History of Chemistry and Chemical Philosophy. Fee, £2.

Courses J. — Applied Chemistry and Metallurgy: Lectures on Technology are given in connection with Laboratory work at hours to be arranged. The subjects are varied in different years. (1) Alkali and other Inorganic Manufactures, including Fuel and Gas. (2) General Principles of Metallurgy. (3) Iron, Steel, and Aluminium. (4) Copper, Lead, Silver, Gold, and other Metals. (5) Technical Gas Analysis.

Course K.—Applied Organic Chemistry.

Course L — Applied Electro-chemistry.

Fee (Courses J, K, L), three terms, £2.

Practical Classes.

(1) Junior. (2) Intermediate: Qualitative Analysis; preparation of Inorganic Compounds; simple Quantitative and Volumetric Analysis. (3) Revision Class. (4) Engineers' Class. (5) General Metallurgy (Elementary).

Chemical Laboratory.

The Chemical Laboratories provide accommodation for every kind of chemical and metallurgical work and research. They include thirty rooms devoted to the study of Chemistry and Metallurgy. Separate rooms are provided for Organic Work, Quantitative Analysis, Water and Gas Analysis, Photometry and Photography, Metallurgy Laboratories, Research Laboratories and Library, and a Museum. Large new Laboratories for Physical Chemistry and Electro-chemistry have been recently erected.

Students desirous of gaining a thorough theoretical and practical acquaintance with Technical Chemistry, or who intend to adopt Chemical work as a profession, must devote three or four years to special study, for which a full curriculum is provided. Fees:—

Per Week.	One Term, Three Months.	Three Terms One Session.
One day	£4	£7
Two days	5 10s.	10
Three days	7	13
Whole week	10 10s.	21

D.P.H. Course (see special syllabus).

Course for Dental Degree (see special syllabus).

Technological Curriculum.

The curriculum extends over three or four years.

The Final Examination for the Associateship of the Institute of Chemistry may be taken after the third year. Those students who have taken the Ordinary Degree of B.Sc. may pass the M.Sc. Exam. in any subsequent year.

The Sheridan Muspratt Scholarship of £50 per annum, tenable for two years, will be competed for in December, 1908, on an Examination in subjects which are included in the first two and a half years of the above curriculum. Candidates should send in their names to the College Secretary not later than November 15. The Sir John Willox Chemical Scholarship, on similar lines, will be open for competition in December, 1909. Other Scholarships, Entrance Scholarships, and Free Student ships are also available to Students.

Evening Classes.

Classes on Metallurgy and Physical Chemistry will be held during the winter.

Prospectuses containing particulars may be obtainep from the Registrar, University of Liverpool.

ARMSTRONG COLLEGE, NEWCASTLE-ON-TYNE.

Professor of Chemistry—P. Phillips Bedson, M.A., D.Sc., F.I.C., F.C.S.

Lecturers in Chemistry—F. C. Garrett, D.Sc., F.C.S., and J. A. Smythe, D.Sc., Ph.D., F.C.S.

Assistant Lecturer and Demonstrator—A. A. Hall, M.Sc., Ph.D.

Lecturer in Agricultural Chemistry—S. Hoare Collins, M.Sc., F.I.C., F.C.S.

Assistant to Lecturer in Agricultural Chemistry—H. Crofts, B.Sc.

First Year Courses.—I. *General Course.*—This Course of Lectures will extend over the three terms of the Session, and is intended to serve as an introduction to the Science. The Lectures will be of an elementary character, and whilst framed to meet the requirements of First Year Students will also be serviceable to such as intend pursuing Chemistry in its various applications in the arts and manufactures, as, for instance, Brewing, Metallurgy, the Manufacture of Soda, Soap, Glass, &c. The subjects treated will include an exposition of the Principles of

Chemistry, and a description of the preparation and properties of the chief Elementary Substances, both metallic and non-metallic, and their more important native and artificial compounds. The class will meet on Mondays, Wednesdays, and Fridays, at 11 a.m., and will commence on Wednesday, October 7th. Fee, £3 10s. for the Session.

II. *Special Course.*—More advanced than the above. Wednesdays and Fridays, 11 to 12. Fee, £3 10s. for Session.

For Diploma in Electrical Engineering.—Tuesday, 12 to 1. A course of Lectures will be given dealing with the Principles of Chemistry, Fuel, the Phenomena of Combustion, and Metals and Alloys employed in Engineering, &c. Fee for the course, £2.

Second Year Courses.—The Lectures for the second year students consist of a course of Lectures on Organic Chemistry, and a course of Lectures on Inorganic Chemistry. The class on Organic Chemistry will meet on Tuesdays and Thursdays at 11 a.m., and will commence on October 6th. The class for Inorganic Chemistry meets at 11 a.m. on Mondays. Fee for Session, £3 10s.; for Inorganic alone £1 10s.; and for Organic alone £3.

Third Year Courses.—Advanced Classes are held for the study of Inorganic, Organic, and Theoretical Chemistry. Fee for the course, £3 10s.

A Lecture Course in Analytical Chemistry will be given on Fridays, at 9.15 a.m. Fee for the course, £1.

Metallurgy and Assaying.—Lecturer, Prof. Louis, M.A., D.Sc., F.I.C., F.G.S.; Demonstrator, H. Dean, A.R.C.M. A Metallurgical Laboratory is provided, in which instruction is given in the ordinary processes of Dry Assaying, and in the preparation and analysis of Alloys, &c. Fees as for Chemical Laboratory.

Agricultural Chemistry.—The instruction in this branch of Chemistry will consist of a series of Lectures and of special practical work in the Chemical Laboratory. Students will be expected to have a knowledge of Elementary Chemistry, such as may be obtained by attending the General Course.

The Lecture Course in Agricultural Chemistry is arranged for two days a week throughout the Session. Fee, £3 10s.

Practical Chemistry.—The Laboratory is open from 10 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it closes at 1 p.m. *Laboratory Fees.*—Students working two days, £3 10s. per term, £9 per session; one day per week, £2 per term, £5 per session.

Courses of Study.—Students will be divided into two classes:—(1) Regular, or Matriculated Students, who are also Members of the University of Durham; and (2) Non-Matriculated Students. Regular Students will be required to follow such a course of study in the subjects professed in the College as will enable them to pass the Examinations for the degree of Bachelor in Science of the University of Durham. Non-Matriculated Students will attend such classes as they may select. Every candidate for admission as a matriculated student must have passed the University Matriculation Examination.

The University has recently adopted the following Matriculation Examination for students entering the Faculty of Science:—Additional Mathematics; English; English History; any *one* subject selected from Religious Knowledge, Botany, Zoology, Physical and General Geography, Experimental Science; any *two* subjects selected from Latin, Greek, French, German. (Students may substitute Extra Mathematics for one of these optional subjects up to and including September, 1911).

In order to enter on a three years' course of study for a Degree in Letters a student must have previously satisfied the Examiners in the following subjects:—Elementary Mathematics or Additional Mathematics; English; English History; either Latin or Greek; either French or German; *one* of the following subjects—Religious Knowledge, Botany, Zoology, Physical and General Geography, Experimental Science.

N.B.—(i.). Students who require to take three subjects to qualify for degrees in Science may offer any three of the following:—Additional Mathematics, Extra Mathematics, English, Latin, Greek, French, German, Physical, and General Geography, English History.

(ii.). Foreign Students may be exempted from the Matriculation Examination on report from the Council of the College of Medicine, or from the Board of Professors of Armstrong College, as the case may be, that they have already passed an examination equivalent to the Matriculation Examination of the University, and that they have sufficient knowledge of English to enable them to follow the courses of instruction they are entering for.

(iii.) Students from Oriental countries may be exempted from the Matriculation Examination on report from the Council of the College of Medicine, or from the Professors of Armstrong College, as the case may be, that they have received such an education in their own country as will enable them to profit by University study, and that they have sufficient knowledge of English to enable them to follow the courses of instruction they are entering for.

For detailed Syllabuses and complete Regulations the College Calendar should be consulted.

Bachelorship in Science.—Every candidate for the Bachelorship in Science will be required to satisfy the examiners in Mathematics, Physics, Chemistry, and either Geology or Natural History—in an examination to be held at the end of the candidate's first year, and at the end of the third year in an examination in one chief and two auxiliary subjects. For details of the subjects the College Calendar should be consulted. Candidates may qualify for the degree of B.Sc. by attending special courses in Agriculture, Engineering, and Mining, and passing the several examinations.

Exhibitions.—Two Exhibitions of the value of £20 and £10 respectively will be awarded in October next to Candidates desirous of attending the first year course of study in the College.

The examination will be held at the College, and will commence on Thursday, October 1st. Candidates should send in their names to the Secretary on or before September 12th.

Evening Lectures.—Courses of Evening Lectures will be given, with a Practical Class for Laboratory instruction.

Several valuable Scholarships are available for students, including the Johnston Chemical Scholarship of the value of £60 for one year, which is open to Bachelors of Science of any British University; the examination for this Scholarship will be held during the week commencing September 28th. Candidates should send in their names to the Secretary on or before September 20th.

THE UNIVERSITY OF MANCHESTER.

Professor of Chemistry and Director of the Inorganic Laboratories—Harold B. Dixon, M.A., M.Sc., F.R.S.

Professor of Chemistry and Director of the Organic Laboratories—W. H. Perkin, Ph.D., F.R.S.

Senior Lecturers and Demonstrators—G. H. Bailey, D.Sc., Ph.D.; J. F. Thorpe, Ph.D., F.R.S.; C. Weizmann, Ph.D.; Norman Smith, D.Sc.

Assistant Lecturers and Demonstrators—E. C. Edgar, D.Sc.; H. F. Coward, M.Sc; Ida Smedley, D Sc.; and J. L. Simonsen, M.Sc.

Professor of Metallurgy—H. C. Carpenter, M.A., Ph.D.

Lecturer in Electro-Chemistry—R. S. Hutton, D.Sc.

The Session begins October 6, 1908.

The Chemical Classes form part of the Courses for Degrees in the University.

Chemistry Lecture Courses.

General Chemistry Course.—Tuesdays, Thursdays, and Saturdays, at 9.30, during the two Winter Terms.

Introduction to Organic Chemistry.—Wednesdays and Fridays, at 9.30, during the Lent Term.

These courses are intended for Students commencing the study of chemistry.

First Year Honours Course.—Mondays, Wednesdays, and Fridays, 11.30, during the two Winter Terms. The Non-Metals.

Second Year Honours Course.—Mondays, Wednesdays, and Fridays, at 2, during the two Winter Terms. The Metals.

Third Year Honours Course.—Theoretical and Physical Chemistry.

Organic Chemistry (General).—Mondays and Fridays, at 9.30, during the two Winter Terms.

Organic Chemistry (Advanced).—Tuesdays and Wednesdays, at 9.30, during the two Winter Terms.

History of Chemistry.—Short Courses during the two Winter Terms.

Metallurgy.—Introductory Course, followed by either—(A) Lead, Copper, Bismuth, Antimony, Zinc, and Tin; or (B) Iron and Steel. Each Course, one Lecture per week during the Session.

Electro-chemistry.—General Theoretical Course: Two hours per week during the Michaelmas Term.

Certificate in Applied Chemistry.—The course extends over three years, and comprises systematic instruction by means of lectures and practical work in the laboratories.

For further particulars of any of these courses apply to the Registrar, Edward Fiddes, M.A.

UNIVERSITY COLLEGE, NOTTINGHAM.

Departments of Chemistry and Metallurgy.

Professor of Chemistry—F. Stanley Kipping, Ph.D., D.Sc., F.I.C., F.R.S.

Demonstrators of Chemistry—R. M. Caven, D.Sc., F.I.C.; G. Sand, D.Sc.; and G. Martin, Ph.D, B.Sc.

The Classes of the College are open to students of both sexes above sixteen years of age.

The Session commences in October, for both Day and Evening Classes.

Lecture Courses.—The Chemistry Day Lectures extend over three years. In the first year a student enters for the course on Elementary Chemistry. In his second year he attends Lectures on both Inorganic and Organic Chemistry. In his third year he attends courses on Advanced Organic Chemistry, Physical Chemistry, and Advanced Inorganic Chemistry.

The fees for the Day Lectures and Classes are as follows: First year, two Lectures per week, 15s. per term. Second year, three Lectures per week, 22s. 6d. per term. Third year, four Lectures per week, 30s. per term.

Demonstrations and Lectures on Analytical Chemistry are given, and Chemical Calculation and Tutorial classes are also held. Various short courses of lectures on special subjects are delivered during the Session.

Students may qualify themselves by attendance at these lectures and classes for the Examinations of the Universities of London, Cambridge, or Oxford, and for the Medical Examinations of the Royal College of Surgeons and of the Universities of Cambridge and Edinburgh: they may also obtain instruction in Chemistry for technical or other purposes, and can enter for a full Chemical Engineering Curriculum. Special attention is given to the requirements of candidates for the Associateship of the Institute of Chemistry.

Practical Chemistry and Metallurgy.—The Chemical and Metallurgical laboratories are open every day from 9 to 5, except on Saturday, when the hours are from 9 to 1; also on Tuesday and Thursday evenings from 7 to 9. Each Student works independently of other Students at a course recommended by the Professor. Instruction is given in general Chemical Manipulation, in Qualitative and Quantitative Analysis, and in the methods of Original Chemical Investigation and Research; Students are also enabled to work out the applications of Chemistry to Pharmacy, Metallurgy, Dyeing, Agriculture, Brewing, Iron and Steel, Tanning, and other Manufacturing Processes. Fees for day students: For one term, £7; for the session, £18; for six hours weekly 40s., and 5s. extra for each additional hour per week. Evening students, 10s. for 2 hours per week, 3 hours 15s., 4 hours 20s., per term.

Research Work.—Students or others wishing to undertake research work in pure or Applied Chemistry will be afforded every facility for doing so and may be admitted at reduced fees. The Laboratories are fully equipped with apparatus and chemicals necessary for such work.

Courses of Technical Chemistry Lectures are also given on Engineering, Dyeing and Bleaching, Brewing, Plumbing, Bread-making, Gas Manufacture, and on other processes of applied Chemistry.

Pharmaceutical Students are provided with Lectures and Laboratory work suitable for the preparation for the Minor and Major Examinations.

Evening Classes.—Evening Lectures and Laboratory instruction will be given in Pure and Applied Chemistry, and the laboratories are open for practical work on Tuesday and Thursday evenings from 7 to 9. Fee for each Lecture Course, 5s.; for each Laboratory Course, 10s.

Full information concerning all College Classes is given in the College Prospectus, price one penny.

THE UNIVERSITY OF SHEFFIELD.

Professor of Chemistry—W. P. Wynne, D.Sc., F.R.S.

Lecturers and Demonstrators—W. E. S. Turner, M.Sc., B.Sc.; W. J. Jarrard, B.Sc.; C. R. Young, B.Sc.

The Session will commence October 7th.

Matriculation Course. — Tuesday and Friday at 9.30. Fee, £2 12s. 6d., and three hours per week Laboratory work.

Candidates for the Intermediate Examination are required to satisfy the Examiners in three of the following subjects:—Pure Mathematics, Applied Mathematics, Physics, Chemistry, and Biology. And those for the Final Examination in three of the following subjects:—Pure Mathematics, Applied Mathematics, Physics, Chemistry, Zoology, Botany, Physiology, Geology, Education.

Intermediate Course in Chemistry for B.Sc. or M.B.—Monday, Tuesday, Thursday, and Friday at 9.30 a.m. £5 5s., and nine hours per week Laboratory work.

B.Sc. Course in Chemistry.—Monday at 9.30 a.m., Tuesday at 5 p.m., and Friday at 2.30 p.m., £4 4s., and 12 hours per week Laboratory work during two Sessions.

B.Sc. with Honours.—Honours Students in Chemistry, after passing the Intermediate Examination, devote the whole of their time to the study of Chemistry, and are expected to reach the standard for a pass in Chemistry for the ordinary degree by the end of the second year. During the second or third year they devote one day a week to lectures and practical work in a subsidiary subject—Mathematics, Physics, or Metallurgy—selected for the degree. The third year is devoted to the advanced study of Chemistry, either chiefly Physical and Inorganic or chiefly Organic, as the student may select. At least four days a week during the second and third years are spent in the laboratory.

M.Sc.—This degree may be conferred upon a Bachelor of Science with Honours who is of one year's standing from the date of his graduation as a Bachelor of Science, or upon any Bachelor of Science who has either passed an examination in an Honours School subsequent to graduation, or has for at least one year after graduation done research work at the University, and has presented a thesis, approved by the Faculty of Pure Science, upon the research work done during that period.

D.Sc.—The degree of Doctor of Science may be conferred upon any Master of Science of not less than five year's standing from the date of his admission to the degree of Bachelor, provided that he has published, in recognised journals or transactions, a research or researches of special merit and approved by the Faculty of Pure Science as qualifying him for the degree.

Laboratory.—Working hours to be arranged between Professor and Students.

Sessional Fees for Day Students:—Three hours per week, £3 3s.; Six £5 5s.; Nine, £7 7s.; Twelve, £9 9s.; Eighteen, £12 12s.; Twenty-four, £14 14s.; Thirty-two,

£16 16s.; Honour or University Students taking eighteen hours or more per week, £12 12s.

Students joining the Laboratory at Christmas will be charged two-thirds and at Easter one-third of the Fees for the whole Session.

A Course of Lectures, with a special class in Laboratory Work, is arranged for Medical Students entering for the Conjoint Board Examinations. Fee, £7 7s.

A course of Elementary Practical Physics and Practical Chemistry, which will meet the requirements of candidates for the Diploma of Public Health is held during the Michaelmas and Lent terms. Fee £5 5s.

An arrangement has been entered into with the Board of Education, South Kensington, which will enable Science Teachers to work in the Chemical Laboratory for three, six, or twelve hours a week on payment of one-quarter of the usual fee, the Board being willing to pay the remainder under certain conditions, of which full information may be obtained on application to the Registrar of the University.

Evening Classes.—Lecture Class and Laboratory, on Wednesday evening during the Michaelmas and Lent terms. Fee, £1 10s.

DEPARTMENT OF METALLURGY.

Professor—J. O. Arnold, D.Met.

Assistant Professor—A. McWilliam, M.Met.A.R.S.M.

This Department has been equipped to meet the requirements of the local industries. The Laboratory is fitted with the most modern apparatus for metallurgical analysis, more especially with appliances for the rapid and accurate chemical examination of Iron and Steel, Fuel, and Refractory materials. It also contains a complete pyrometric installation, and a laboratory for the study of the micrographic analysis of metals fully equipped with specially designed microscopes by Ross, polishing tables, etching appliances, incandescent light for evening work, &c. The School is now the most complete of its kind for teaching the practical manufacture, the chemical constitution, and the physical properties of Steel. Special attention is given to the determination of the microscopic constituents of Steel. Although the chief industry of the district occupies the central position in the course of instruction, general metallurgy is not neglected, but is dealt with in a separate syllabus, dealing with metals (other than iron and steel) used in the arts. Students are thus enabled to select and at once enter upon a course of scientific metallurgical training of immediate practical utility. They may take up and work through any portions of the course, but certificates will be granted only to those who follow the prescribed courses, and pass the necessary examinations. Lectures on Iron and Steel Manufacture, on Fuel and Refractory Materials, and on General Metallurgy. Practical Metallurgy:—Laboratory, Furnaces, Foundry, and Testing Machine Course; Practical Course of Metallurgy other than Iron and Steel; Practical Fuel Course for Students in Collieries or Gas Works.

A new steel works has recently been erected in connection with the Sheffield College at a cost of about £15,000. The works include a two ton open-hearth furnace, a one ton Bessemer plant, and a four-hole crucible melting plant. The works is also provided with a hammer capable of dealing with four-inch ingots Differential annealing furnaces and gas and electric hardening furnaces constitute part of the new plant. Additional laboratory accommodation is attached to the works, viz., chemical laboratory for staff, chemical laboratory for post graduate students, together with a complete magnetic laboratory for hysteresis, &c.

UNIVERSITY COLLEGE, DUNDEE.

UNIVERSITY OF ST. ANDREWS.

Professor of Chemistry—(Vacant).

Assistant Lecturers—J. K. Wood, D.Sc., and T. Blackadder, B.Sc.

Lecture Assistant and Lab. Steward—J. Foggie, F.C.S.

The Winter Session begins early in October and ends in March. The Summer Session extends from the middle of April to the end of June.

The Junior Lecture Course on Systematic Chemistry is given daily during the Winter Session, and embraces the Elements of Inorganic and of Organic Chemistry.

Advanced Courses, of about fifty lectures each, will be given during the year as follows:—

Organic Chemistry; Inorganic Chemistry, including the more important technological applications; Theoretical and Physical Chemistry; Bleaching and Dyeing, including the Chemistry of the Textile Fibres.

Practical Instruction in all of the above branches will be given in the Laboratories and Dye-house. Special facilities are afforded to Research Students.

UNIVERSITY OF ABERDEEN.

CHEMISTRY.

Professor—Francis R. Japp, M.A., LL.D., F.R.S.

Demonstrators—Francis W. Gray, M.A., B.Sc., and Joseph Knox. D.Sc.

I. *General Lecture Course* (100 *Lectures*).—Daily during the Winter Session. The subjects treated of include (1) The Laws of Chemical Combination and the General Principles of Chemistry; (2) Non-metallic and Metallic Elements, and their Compounds; (3) Organic Chemistry; (4) Applications of Chemistry to the Arts and Manufactures. Fees, for first attendance, £4 4s.; for subsequent attendance, £3 3s. A Tutorial Class (without fee), conducted by the Junior Demonstrator, is held in connection with this course.

II. *Special Lecture Course on Organic Chemistry* (50 *Lectures*).—Daily during the Summer Session. Fee, £3 3s.

III. *Practical Course for Medical Students.*—This course, which occupies five hours a week during the Summer Session—in all fifty hours—is devoted to practice in Elementary Qualitative Analysis. Fee, £3 3s.

IV. *Chemical Laboratory.*—The Laboratory is open to Students daily from 9 a.m. to 5 p.m. Each Student on entering is allowed to arrange his hours of work so as to suit his own convenience, but must adhere to these hours when once fixed. The Laboratory instruction includes: (1) General experiments; (2) Preparations; (3) Qualitative analysis; (4) Quantitative analysis: gravimetric, volumetric, and gasometric. The Course qualifies for the examinations of the Institute of Chemistry. Fee, £3 3s. a session. A certain number of free places are available, on the recommendation of the Professor, and subject to the approval of the Senatus, for research students.

V. *Physical Chemistry* (60 *Lectures*) —In connection with the Laboratory work, three Lectures a week on Physical Chemistry are delivered during the Winter Session by the Lecturer, Mr. F. W. Gray. Fee, £2 2s.

UNIVERSITY OF ABERDEEN AND ABERDEEN AND NORTH OF SCOTLAND COLLEGE OF AGRICULTURE.

Agricultural Chemistry.

Lecturer—James Hendrick, B.Sc., F.I.C.

Assistants—R. Glegg, B.Sc., F.I.C.; W. J. Profeit, M.A., B.Sc.; and John Macpherson, M.A., B.Sc.

I. Preparatory Courses in General Chemistry and in Organic Chemistry are given for those who are unable to take the full University Courses in these subjects. The course in General Chemistry consists of about sixty Lectures with Practical Work (one hour daily) during the Winter Session. The course deals with the general principles of Chemistry, and with those parts of Inorganic Chemistry which are of more immediate concern to students of Agricultural Chemistry. The Practical Work goes along with and illustrates the Lectures. Fee, £4 4s.

The course in Organic Chemistry consists of about thirty Lectures, and is given during the Winter Session. It deals especially with those parts of the subject which are directly related to Agricultural Chemistry. Fee, £1 1s.

II. *Agricultural Chemistry* (*General Course*).—This class meets daily during the Winter Session, and includes both Lectures and Laboratory Work. The Lectures deal with the Chemistry of the Atmosphere, the Soil, Manures, Foods, Preservatives, Insecticides, &c. The Physiological Chemistry of Plants and Animals, the Composition and

Manurial Requirements of Crops, and Dairy Chemistry are also treated of. The Laboratory Work is primarily intended to accompany and illustrate the Lectures. Exercises dealing with the properties and composition of Soils, Manures, Feeding-stuffs and Waters, and with the impurities and adulterations of these, occupy much of the time given to Practical Work. The fee for the whole Course (Lectures and Practical Work) is £4 4s.

III. *Laboratory.*—A Summer Course in Practical Chemistry is given to prepare students who have not previously done sufficient laboratory work for the course in Agricultural Chemistry. Fee, £2 2s.

IV. The Laboratory is open daily from 9 a.m. to 5 p.m. for students who wish to study the principles and practice of Agricultural Chemistry or to undertake research in this subject.

UNIVERSITY OF EDINBURGH.

Department of Chemistry.

Professor—James Walker. D.Sc., Ph.D., F.R.S.

Lecturers—L. Dobbin, Ph.D.; H. Marshall, D.Sc., F.R.S.; and W. W. Taylor, M.A., D.Sc.

The working terms are—Winter Session, from beginning of October to middle of March; Summer Session, from beginning of May to middle of July.

Lecture Courses.—During the Winter Session a General Course of Chemistry for medical, arts, and science students is given by the Professor. The class meets daily; fee £4 4s. Advanced Courses of 25 lectures each are given in the Winter and Summer Sessions; fee £1 1s. An Intermediate Course of Organic and Advanced Inorganic Chemistry is held in summer; fee, £2 2s. There is also a class on History of Chemistry or Chemical Theory, by Dr. Dobbin; fee £1 1s.: and a class on Mineralogy and Crystallography, by Dr. Marshall; fee, £2 2s. All these Lectures, except the General Course, are now open to women, and a separate General Course for women in the faculties of Arts and Sciences has been arranged for the forthcoming Winter Session.

In addition to the above, Lecture Courses are from time to time given by the Assistants on particular branches of Organic and Inorganic Chemistry. These Lectures are free to Laboratory Students.

Laboratories.—Practical classes for Medical Students meet during the Winter Session and in the Summer Session. (Fee, £3 3s.) The laboratories for Science and Arts Students engaged in analytical and advanced practical work are open daily from 9.30 till 4.30. (Fees: Whole Day—Winter Session, £10 10s.; Oct.-Dec., Jan.-March, or Summer Session, £5 5s. Half Day—Winter Session, £6 6s.; Oct.-Dec., Jan.-March, or Summer Session, £3 3s. Preference will be given to students in the above order). Full Courses of instruction are given in Analytical, Practical Organic and Inorganic Chemistry (including Gas Analysis), Physico-chemical Measurements, Agricultural Chemistry, and Assaying. Facilities are afforded to advanced students who desire to undertake chemical investigations.

Various prizes and scholarships are attached to the laboratory and general class.

Graduation.—Two Degrees in Pure Science are conferred, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.).

Candidates for Degrees in Science, if not graduates (by examination) in Arts in one of the Universities of the United Kingdom or in a Colonial or Foreign University recognised for the purpose by the University Court, must pass a preliminary examination in (1) English; (2) Latin, Greek, French, or German; (3) Mathematics; (4) One of the languages Latin, Greek, French, German, Italian, not already taken under (2), or Dynamics. In the case of a student whose native language is other than European, the Senatus may, at the Preliminary Examination, accept such language as a substitute for a modern European language. The Senatus may also in such a case accept as an alternative to Latin or Greek any other classical language, such as Sanscrit or Arabic.

The First B.Sc. Examination embraces Mathematics, or Biology (*i.e.*, Zoology and Botany), Natural Philosophy, and Chemistry. The Final B.Sc. Examination in Pure Science includes any three or more of the following subjects:—Mathematics, Natural Philosophy, Astronomy, Chemistry, Human Anatomy (including Anthropology), Physiology, Geology (including Mineralogy), Zoology (including Comparative Anatomy), and Botany (including Vegetable Physiology). In the Final Examination two written papers are set in each subject professed, the second of a higher standard than the first. Candidates must pass the first section in all, and the second section in at least one, of the subjects professed; the same regulations apply also to the Practical and Oral Examinations. Chemistry in this examination embraces Inorganic, including Mineralogical, Chemistry; Organic Chemistry; Physical Chemistry; Chemical Crystallography; History of Chemistry. In the written papers a choice of questions is allowed, so as to adapt the examination to the various courses of advanced study which candidates may have selected. Practical Examination:—Complex Qualitative Analysis; Inorganic Preparations; Gravimetric and Volumetric Analysis. Each candidate taking the higher standard will also be examined on Organic Preparations, Ultimate Organic Analysis, and one of the following subjects, selected by himself:—Gas Analysis; Assaying; Physico-chemical Measurements, including Practical Crystallography as a special branch.

A candidate for the D.Sc. Degree must submit a thesis on original work done by him. The Thesis must be approved before the candidate is allowed to proceed to Examination. The candidate in Chemistry *may be* required to pass a searching examination in one of the following branches:—(1) The Chemistry and Chemical Technology of Inorganic Bodies, including Metallurgy; (2) Organic Chemistry; and to show a thorough practical acquaintance with chemical analysis in all its branches, and with the preparation of pure substances.

HERIOT-WATT COLLEGE, EDINBURGH.

Principal—A. P. Laurie, M.A., D.Sc., F.R.S.E.

Professor—John Gibson, Ph.D., F.R.S.E.

Assisted by the following Lecturers and Demonstrators:—R. B. Denison, D.Sc., Ph.D.; Archibald Boon, B.A., B.Sc.; Andrew F. King, F.I.C.; E. B. R. Prideaux, M.A., D.Sc.

The Session begins October 1st, 1908.

The curriculum of this College comprises both Day and Evening Classes, each department providing the higher general and technical education.

The Chemistry Course is designed to meet the wants of Manufacturing Chemists. The instruction consists of Courses in Chemistry, Physics, Mathematics, Engineering, and Mechanical Drawing, with Special Courses in Gas and Paper Manufacture, Brewing, &c., by Experts, in the fourth year. Special attention will be paid to Electrochemistry. The Chemistry Classes are recognised by the University as qualifying for the B.Sc., in Chemistry, and are also recognised by the Institute of Chemistry.

For Students who have been three and four years in the Chemistry Department, arrangements have been made for their spending from four to twelve months in the laboratories of the Corporation's Gas Works, with a view to the study of problems connected with the Combustion of Fuel, the Manufacture of Coal-gas and its By-products, &c.

A Laboratory, under the charge of Dr. Emil Westergaard, has been provided for the study of Technical Mycology, including Brewing, Distilling, Malting, Vinegar Making, Tanning, Preserving, Starch and Sugar Making, Bread Making, Butter and Cheese Manufacture, &c.

Matriculation Fee, 5s.; Composition Fees from £12 12s. to £15 15s. Full particulars are published in the Calendar of the College, which is issued early in September.

GLASGOW AND WEST OF SCOTLAND TECHNICAL COLLEGE.

Professor of Chemistry—G. G. Henderson, D.Sc., M.A.
Professor of Technical Chemistry—T. Gray, D.Sc., Ph.D.
Professor of Metallurgy—A. Humboldt Sexton, F.C.S., F.R.S.E.

Also, Professors and Lecturers in the other leading branches of Pure and Applied Science and Technology.

Session opens Sept. 21.

The main objects of this College are to afford a suitable education to those who wish to qualify themselves for following an industrial profession or trade, and to train teachers for technical schools. It was founded by an Order in Council, dated 26th November, 1886, according to a scheme framed by the Commissioners appointed under the provisions of the Educational Endowments (Scotland) Act, whereby Anderson's College, the Young Chair of Technical Chemistry in connection with Anderson's College, the College of Science and Arts, Allan Glen's Institution, and the Atkinson Institution were placed under the management of one governing body.

The Diploma of the College is awarded to Day Students who have attended prescribed courses of instruction and passed the necessary examinations. The ordinary courses extend over three years, but arrangements are made for advanced students continuing their studies in special departments. The diploma course in Chemistry extends over four years.

Complete courses of instruction are provided in both Day and Evening Classes.

Copies of the Calendar for 1908-1909 may be had from Mr. H. F. Stockdale, Secretary; price by post, 1s. 4d. Prospectuses will be sent free.

UNIVERSITY OF ST. ANDREWS.

UNITED COLLEGE OF ST. LEONARD AND ST. SALVATOR.

Professor of Chemistry — T. Purdie, B.Sc., Ph.D., LL.D., F.R.S.

The Session begins on October 7th. A Competitive Examination, open to intending Students of Arts, Science, and Medicine, for about thirty-five Bursaries, ranging in value from £40 to £10 each per annum, will be held on September 11th and following days. Nineteen of these Bursaries are restricted to Men, two are open to Men or Women, and fourteen are restricted to Women, the latter being intended for women who at the conclusion of their Arts or Science Course will proceed to Medicine.

A Hall of Residence is provided for Women Students.

Two Degrees in Science are conferred by the University of St. Andrews, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.), and Chemistry is also included in the curriculum for the M.A. Degree; the regulations will be found in the "University Calendar."

Lecture Courses.

Two distinct Courses of Lectures are given, each comprising at least one hundred meetings of the class.

First Year's Course.—This Class meets at 11 o'clock on five days in the week. The introductory lectures treat of the Nature of Chemical Action, the Classification of Substances into Elements and Compounds, the Phenomena of Oxidation, and the Composition of Air and Water. The Laws of Chemical Combination and the Atomic Theory are next discussed, after which the more commonly occurring elements and inorganic compounds are described systematically. Elementary Organic Chemistry is also included in the Course.

The chemistry of manufactures is referred to only cursorily; special attention, on the other hand, is given to those parts of the science which are of general educational value, and as much of the theory of chemistry is introduced as is compatible with elementary treatment.

This course of instruction is intended to meet the requirements of the M.A., First Science, and M.B., Ch.B. Exams., so far as Theoretical Chemistry is concerned.

Second Year's Course.—The first part of the Course is devoted to Organic Chemistry, and the second part to General and Physical Chemistry, the instruction in general being such as is required for the Final Science Exam.

Certificates are awarded on the results of examinations, and the "Forrester Prize" of about £10 is awarded to the best Student of the year.

Fee for the Session, for each Course, £4 4s.

Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., except on Saturdays, when it is closed at 1 p.m. The work pursued in the Laboratory comprises:—(1) The performance of experiments illustrative of the Principles of Inorganic and Organic Chemistry; (2) Qualitative and Quantitative Analysis. Each student pursues an independent course of study under the supervision of the Professor or Demonstrator, the nature of the work varying with the proficiency of the student and the particular object he may have in view. Suitable courses of instruction in Practical Chemistry are provided for candidates for the Examinations in Arts, Science, and Medicine.

The fee for the Ordinary Course of Practical Chemistry is £3 3s. and for the Advanced Course £5 5s.

A special Research Department has been instituted by the University, the laboratories of which are open to students who give proof of their capacity of conducting chemical investigation.

QUEEN'S COLLEGE, BELFAST.

Professor—E. A. Letts, Ph.D., D.Sc., F.R.U.I., F.R.S.E., &c.

I.—*Chemistry.*—The lectures are delivered at 3 p.m., on the first five days of each week, and terminate at the end of March The course is divided into three parts:—(1) Chemical Philosophy; (2) Inorganic Chemistry; (3) Organic Chemistry. Fee, £2.

II.—*Practical Chemistry.*—In this course the Students are instructed in the general methods of conducting Chemical Analyses. Fee, £3.

III.—*Laboratory Pupils.*—The Chemical Laboratory is open from November until the end of March, and from May 1st until the third week of July, on the first five days of the week, from 10 a.m. until 4 p.m. Students are admitted as working pupils on payment of a fee of £5 for the first period, or of £3 10s. for the second period (or for a single term).

The new Chemical Laboratories are now open, and are provided with all modern appliances. Special facilities are offered to those engaged in Research work.

Scholarships.—The following College Scholarships are awarded specially in connection with the schools of Chemistry and Physics:—Senior Scholarships in Chemistry alone awarded annually of £40, and tenable for one year. Andrews Studentship in Chemistry and Physics, value about £80 annually, and tenable for two years. 1851 Exhibition Scholarships: One of these Scholarships has hitherto been placed at the disposal of the College every two years, of the annual value of £150. tenable for two or under special conditions for three years.

The following Scholarships, &c., of the Royal University of Ireland are awarded in the same group of subjects (Chemistry and Physics):—At the B.A. Degree examination, First-class Exhibitions of £42 each and Second-class Exhibitions of £21. At the M.A. Degree examination, a Studentship of £100 per annum, tenable for three consecutive years. Also a Junior Fellowship, tenable for four years, of the annual value of £200.

QUEEN'S COLLEGE, CORK.

Professor—Augustus Edward Dixon, M.D.
Demonstrator—John Taylor, M.Sc.

The College Session will commence in October, 1908, and end in June, 1909. All classes are open to male and female students.

The courses in Chemistry, though designed primarily to meet the requirements of candidates proceeding to the Examinations in Arts, Medicine, and Engineering of the Royal University of Ireland, of the Medical Licensing Bodies of Dublin and Edinburgh, and of the Pharmaceutical Society of Ireland, can be arranged specially as required, so as to render them suitable to any particular course of study. The following courses are provided:—

Systematic Chemistry.

I. General Lecture Course; three terms. Inorganic Chemistry, Elementary Organic Chemistry, and Chemical Philosophy.

II. Advanced Organic Chemistry, and Chemical Philosophy.

Practical Chemistry.

III. Winter Course of Practical Analytical Chemistry; commencing early in January, 1908.

IV. Summer Course of three months' duration, for students of Medicine; ending about the third week in June.

V. Course for Pharmaceutical Students; held during the second and third terms.

Laboratory Work.

VI. The Chemical Laboratory is open daily from 10 to 4 o'clock, except on Saturdays, to Students entering for Special Courses of practical work in General Inorganic Chemistry, Qualitative and Quantitative Analysis, Organic Chemistry, or for the purpose of Original Investigation.

Scholarships, &c.—In addition to the Class Prizes given at the Sessional Examinations, the College awards a Senior Scholarship of the value of £40, for Chemistry and Physics (either of which may be taken as a limited course), and numerous Junior Scholarships in Arts, Medicine, and Engineering, of which chemistry forms a part, value from £20 to £24 each, annually. The Royal University of Ireland also offers Exhibitions in Chemistry and Physics, First-class value £42, and Second-class value £21, together with other prizes, *e.g.*, a Studentship of £100 per annum, tenable for three years; and the 1851 Exhibition Scholarships, value £150, tenable for two, and, under certain circumstances, for three years, have from time to time been placed at the disposal of the College.

Full particulars as to Lectures, Fees, Scholarships, Exhibitions, &c., are contained in the Regulations extracted from the College Calendar, which will be supplied on application to the Registrar.

QUEEN'S COLLEGE, GALWAY.

Professor—Alfred Senier, Ph.D., M.D., F.I.C., F.C.S., M.R.I.A.

Demonstrator—Rosalind Clarke, B.A.

Research Assistant—Frederick G. Shepheard, B.Sc., A.R.C.S., F.C.S.

The Session commences in October and ends in June.

Chemistry is studied by attendance at Lectures, by work in the Laboratories, and by the use of the College Library. The Courses in the several faculties are arranged with a view to the requirements of the Royal University of Ireland, but are adapted also to those of other Universities and licensing bodies.

Lecture Courses. Faculty of Arts. — 1. Second year's Course, Inorganic and Organic, and the Elements of General Chemistry. 2. Third year's Course, Advanced Organic Chemistry. 3. Fourth year's Post-Graduate Course, Advanced General Inorganic and Organic Chemistry. *Faculty of Medicine.*—First year's Course, Inorganic and Elementary Organic Chemistry. *School of Engineering.*—First year's Course, Inorganic Chemistry.

Laboratory Courses. Faculty of Arts. — 1. Second year's Course, Exercises in Inorganic Qualitative Analysis. 2. Third year's Course, Quantitative Analysis and other experiments to suit the requirements of individual Students. 3. Fourth year's Post-Graduate Course, Advanced Quantitative Analysis, Organic and Inorganic Preparations, and determination of their Physical and Chemical characters. 4. The Laboratories are also open to Students for work in other branches of Chemistry. *Faculty of Medicine.* — 1. Second year's Course, Inorganic and Organic Elementary Qualitative Analysis, and the Chemical Examination of Urine. *School of Engineering.* —1. Second year's Course, Inorganic Qualitative Analysis.

A Scholarship in Chemistry is offered for competition each year of the value of £40, and Chemistry enters into the subjects required for numerous Scholarships in Arts, Medicine, and Engineering. The Royal Commissioners for the Exhibition of 1851 offer every two years a Science Research Scholarship of £150 per annum. This Scholarship has already been held by six Chemistry Students of this College.

For details as to Fees, Regulations as to Scholarships, and other particulars apply to the Registrar, from whom the Calendar, published in December, and the Extracts from Calendar, published in advance in July, may be obtained.

ROYAL COLLEGE OF SCIENCE, DUBLIN.

Dean of Faculty and Professor of Chemistry—W. N. Hartley, F.R.S., D.Sc.

Lecturer on Organic Chemistry—Alphonsus O'Farrelly, M.A.

Senior Assistant—J. Holms Pollok, D.Sc.

Junior Assistant—J. Ivon Graham, A.R.C.Sc.I.

The Royal College of Science supplies, as far as practicable, a complete course of instruction in Science applicable to the Industrial Arts, and is intended also to aid in the instruction of teachers for the local Schools of Science.

Diplomas are awarded in the Faculties of Engineering (Mechanical and Electrical), Applied Chemistry, and Agriculture. If accompanied by a certificate from the Professor of Chemistry, the Diploma of Associate of the Royal College of Science in the Faculty of Applied Chemistry is recognised by the Council of the Institute of Chemistry of Great Britain and Ireland as qualifying candidates for admission to the practical examinations of the Institute.

The instruction in Chemical Science includes (1) General Chemistry; (2) Organic Chemistry, Elementary and Advanced; (3) Analytical and Experimental Chemistry; (4) Instruction in Chemical Research.

Fees payable by Non-Associate Students:—£2 for each separate Course of Lectures. For Analytical Chemistry and Research—£5 for three months; £9 for six months; £12 for the entire session. For Assaying—£5 for three months; £9 for six months. £12 for the entire session.

There are four Royal Scholarships of the value of £50 each yearly, with Free Education, including Laboratory Instruction, tenable for two years; two become vacant each year; they are awarded on the results of their examinations to Associate Students, not being Royal Exhibitioners, who have been a year in the College.

A limited number of Scholarships in Science and Technology are competed for each year. These Scholarships include free education for the full Associateseip Course of three years and maintenance allowance of a guinea a week during the Session.

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THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.—The Institute of Chemistry was founded in October, 1877, and incorporated by Royal Charter in June, 1885, (i.) to promote the better education of persons desirous of becoming professional consulting and technological chemists, public analysts, and chemical advisers; (ii.) to examine Candidates, and to grant certificates of competency; and (iii.) to elevate the profession of Consulting and Analytical Chemistry by setting up a high standard of scientific and practical proficiency and by insisting on the observance of strict rules in regard to professional conduct. *The Studentship.*—Every Candidate for admission to the Studentship is required to produce evidence that he is at least seventeen years of age, and that

he has passed a Preliminary Examination in subjects of general education, recognised by the Council of the Institute. He must also show that, at the time of making application for registration, he is working at a College or University recognised by the Council, or under the direction of a Fellow of the Institute in an approved laboratory, with the object of adopting the profession of Analytical and Consulting Chemistry. *The Intermediate Examination in General Theoretical and Practical Chemistry.*—Candidates for admission to the Intermediate Examination are required to produce evidence (I.) of having passed an approved Preliminary Examination in subjects of general education; (II.) of having regularly attended systematic day courses, in a College or University recognised by the Council, during at least three academic years, in theoretical and practical Chemistry, and courses in Physics, Mathematics, and one of the following subjects, in accordance with the Regulations of the Institute: (i.) Higher Physics; (ii.) Advanced Mathematics; (iii.) Mechanics and Chemical Engineering; (iv.) Metallurgy; (v.) Geology and Mineralogy; (vi.) Physiology; (vii.) Bacteriology; (viii.) Agriculture; (ix.) Elementary Botany; (x.) Elementary Biology; and (III.) of having satisfactorily passed the Class Examinations in all the subjects required to be taken. As an alternative in the matter of training (II.), a candidate may take two years' training in a recognised Institution as indicated above, and work systematically for two other years under a Fellow of the Institute in an approved laboratory. A Candidate who has taken a Degree in Science (including Inorganic and Organic Chemistry, and Physics in the Degree Examination. Mathematics having been also passed in at either the Final or Intermediate University Examination) in a University recognised by the Council, is eligible for admission to the Intermediate Examination of the Institute. Holders of certain Honours Degrees or Diplomas are exempt from passing the Intermediate, and, by virtue of their qualifications, are eligible for admission to the Final Examination direct. *The Final Examination for the Associateship (A.I.C.).*—This comprises, in addition to a general knowledge of all branches of chemistry, a thorough knowledge of one branch—mineral chemistry; metallurgical chemistry, physical chemistry, organic chemistry, analysis of food and drugs and water (including a compulsory examination in therapeutics, pharmacology, and microscopy), or biological chemistry. Any Candidate who has passed the Intermediate Examination of the Institute, or who is entitled to claim exemption from passing the Intermediate Examination, is eligible for admission to the Final Examination. *Fellowship (F.I.C.).*—For admission to the Fellowship, an Associate is required to have been registered for three years, and to have been continuously engaged during that period in the study and practical work of Applied Chemistry in a manner satisfactory to the Council. Full particulars are given in "The Book of Regulations for the Admission of Students, Associates, and Fellows," which may be obtained (Price One Shilling) from the Registrar, 30, Bloomsbury Square, London, W.C.

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City and Guilds of London Institute for the Advancement of Technical Education.—The operations of the City and Guilds of London Institute are divided broadly into four branches: the educational work of three London Colleges, and of the Technological Examinations. Programmes of the London Colleges may be had on application to the Head Office of the Institute, Gresham College, Basinghall Street, London, E.C., or from the respective Colleges. The Technological Examinations (Examinations Department, Exhibition Road, S.W.), are conducted once every year at various centres throughout the kingdom. The Programme of the College, containing full particulars of the conditions of entrance, fees, and courses of instruction, may be had on application. *City and Guilds Central Technical College, Exhibition Road.*—Professor of Chemistry, H. E. Armstrong, Ph.D., F.R.S. The object of this Institution is to give to London a College for the higher technical education, in which advanced instruction shall be provided in those kinds of knowledge which bear upon the different branches of productive industry, whether Manufactures or Arts. The main purpose of the instruction given is to practically demonstrate the application of different branches of science to various manufacturing industries. In order that this instruction may be efficiently carried out, the Institution, in addition to the lecture theatres and class rooms, is fitted with laboratories, drawing offices, and workshops; and opportunities are afforded for the prosecution of original research, with the object of the more thorough training of the students, and for the elucidation of the theory of industrial processes. The courses of instruction are arranged to suit the requirements of—1. Persons who are training to become Technical Teachers; 2. Persons who are preparing to enter Engineers' or Architects' offices, or Manufacturing works; 3. Persons who desire to acquaint themselves with the scientific principles underlying the particular branch of industry in which they are engaged. The Matriculation Examinations begin on Tuesday, September 22nd, and the Winter Session opens on Tuesday, September 29th. *City and Guilds Technical College, Finsbury.*—Professor of Chemistry, Raphael Meldola, F.R.S. The operations of the Technical College, Finsbury, are divided into two distinct portions: Day Classes for those who are able to devote one, two, or three years to systematic technical education; Evening Classes for those who are engaged in industrial or commercial occupations in the daytime and who desire to receive supplementary instruction in the application of Science and of Art to the trades and manufactures in which they are concerned or employed. Each Professor is assisted by Demonstrators. Besides these there are Lecturers and Teachers in special subjects. An examination for the admission of Students will be held at the College on Tuesday, September 22nd. Session begins September 29th. *South London Technical Art School.*—Classes in Modelling, Design, Drawing and Painting, House Decoration.

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Leeds: Central Technical School, Leeds Institute, Cookridge Street, Leeds.—Head Master, R. E. Barnett, B.Sc. (Lond.), A.R.C.S. Evening Classes are held in all subjects of Science and Technology, among which are:—Chemistry (Inorganic and Organic), Chemical Calculations and Principles of Analysis, by Mr. R E. Barnett, B.Sc., A.R.C.S., Mr. J. B. Murray, Mr. A. McFarlane, and Mr. O. F. Kirby, M.A., B.Sc.; Metallurgy (Theoretical and Practical) and Iron and Steel Manufacture, by Mr. B. A. Burrell, F.I.C., F.C.S.; Gas Manufacture, by Mr. W. E. Pettigrew; Oils and Fats, by Mr. W. Dillon; Magnetism and Electricity, Practical Physics, &c., by Mr. J. E. Tindall, B.A., B.Sc.; Bread-making and Flour Confectionery, by Messrs. W. H. Quinn and J. C Hasselgrave; Photography, by Mr. S. E. Bottomley. Fees: from 7s. 6d. per session. Group courses arranged for Students engaged in Chemical Industries. Special three evening Course for Pharmaceutical Students, covering the "Minor" syllabus in three years, by Messrs. J. H. Gough, Ph.C., F.C.S.; S. Parrish, B.Sc., A.R.C.S; and N. Walker. Session commenced Sept. 14th. See the Technical Handbook, 2d. (by post 4½d.). Prospectuses of various Classes free on application to the Head Master, or to Mr. James Graham, Secretary for Education, Calverley Street, Leeds.

City of Leeds Higher Education Department.—Courses of Evening Classes have been arranged for the Minor Examination of the Pharmaceutical Society, in which the whole Syllabus will be covered in three Winter Sessions. Chemistry: Mr. S. Parrish, B.Sc., A.R.C.S., Chief Lecturer in Chemistry, Central High School, and Mr. J. B. Murray. Botany: Mr. Norman Walker, Assistant Lecturer in Botany, Leeds University. Pharmaceutics: Mr. J. H. Gough, Ph.C., F.C.S., Demonstrator in Materia Medica and Pharmacy, Leeds University. Full particulars from Mr. James Graham, Secretary for Education, Education Offices, Leeds.

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Iron and Steel Institute.—In addition to the papers already announced for reading at the Middlesbrough Meeting of the Iron and Steel Institute on September 29 to October 1, a paper will be presented by Mr. William Hawdon (Middlesbrough) on "The Progress in the Cleveland Iron and Steel Industries during the past Quarter of a Century," that is, since the previous visit of the Institute to Middlesbrough in 1883. The paper promised by Mr. J. E. Stead and Mr. T. Westgarth will be held over until the next meeting.

The Chemical Laboratory Fresenius at Wiesbaden.—During the Summer Term, 1908, the Chemical Laboratory Fresenius was attended by twenty-five students. Of these eleven were from Germany, four from Russia, three from the United States of America, two from England, one from Austria, one from Switzerland, one from Sweden, one from Norway, and one from East India. There were two assistants in the Instruction Laboratory, and twenty-two in the Versuchsstationen (Private Laboratories). To the certified staff of teachers belong the Directors, Geh. Regierungsrat Prof. Dr. H. Fresenius, Prof. Dr. W. Fresenius, Prof. Dr. E. Hintz, and also Prof. Dr. med. G. Frank, Dr. L. Grünhut, Dr. R. Fresenius, and J. Huber (Architect). The next Winter Term begins on October 15th. Ladies are also admitted as students. During the past Summer Term, besides the scientific researches, numerous analyses were undertaken in the different departments of the Laboratory (Versuchsstationen) on behalf of trade, manufacture, industry, mining, agriculture, hygiene, justice, and government.

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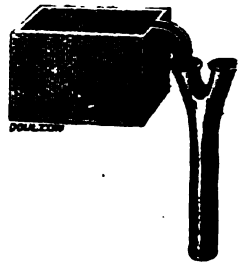

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LITHIUM IN RADIO-ACTIVE MINERALS.*

By Prof. W. N. HARTLEY, F.R.S., Royal College of Science, Dublin.

The question whether lithium is, or is not, a widely occurring element in nature, and whether it is found associated with any other element, more particularly with copper than with the alkalis or the alkaline earths, arises from the assumed transmutation of copper contained in solutions which were exposed to the radiations of radium into lithium, neon, and possibly other substances.

In the course of a discussion on the subject it has been stated by Sir William Ramsay (*Nature*, March 5th, 1908, p. 412):—As sodium and potassium are much more widely distributed than lithium, it is more likely that they are the chief products from copper, and that some modifying circumstance has determined the formation of a trace of lithium. Lithium was mentioned because it is an unlikely constituent of dust, glass, copper, &c., which were tested specially to prove its absence."

There are two distinct statements here which, according to wide experience, appear to require modification.

First, is it the fact that sodium and potassium are much more widely distributed than lithium?

They appear to be more abundantly distributed, but that is not the question. Let us take, first, the evidence of Kirchhoff and Bunsen on this point (*Pogg. Ann.*, 1860, cx., 161).

It was experimentally proved that the eye can detect 9/1,000,000 of a mgrm. of lithium carbonate suspended in the air of a room in which a Bunsen flame was burning, the quantity of sodium chloride perceived in similar circumstances was 1/3,000,000 of a mgrm.; in other words, the sodium chloride flame reaction is twenty-seven times as delicate as that of lithium carbonate.

If these figures be taken for calculating the quantity of lithium and sodium respectively which colour the flame, it will be found that the proportions are approximately 1/600,000 of a mgrm. and 1/1,200,000; so that the reaction for sodium is at least twice as sensitive as that for lithium. The eye is, however, much less sensitive to the red line of lithium than to the yellow line of sodium when the two substances are mixed.

Kirchoff and Bunsen state that lithium is one of the most widely distributed elements in nature, and proceed to substantiate this view. Thus, the presence of lithium was easily detected in 40 cc. of sea-water taken in long. 39° 14′ W. and lat. 41° 41′ N.; in the ashes of seaweed carried by the Gulf Stream to the coast of Scotland; in all varieties of orthoclase and quartz from the granites of the Odenwald; in spring-water from the eastern slopes of the Neckar, near Heidelberg. The laboratory water supply did not contain it, because it came from a different geological source. It was found in the ashes of all kinds of wood, the trees of which grow on a granitic soil; it was found also in varieties of potash from Russia and elsewhere; in the ashes of tobacco, and the leaves and tendrils of the vine; in the ashes of cereals of all kinds grown on granitic soil on the plain of the Rhine; in the blood and muscle of ruminant animals, and likewise of man. Subsequently, it was detected in a few decigrms. of the solid residue of sea-water,† and in many mineral waters, in different varieties of marble, and in many calcareous rocks, also in English chalk. Traces of lithium were also found in potash-glass.

More recently, evidence has been derived from facts which may be divided into three categories. First, those observed in the spectroscopic and spectrographic analysis of common ores and minerals from different parts of the world, and such as are not associated with the alkali metals or with radio-active substances. Mr. Ramage and I examined more than 170 of these substances, and without exception they all contained sodium and potassium, and the potassium almost invariably contained lithium; but a more remarkable fact was brought to light by the detection of rubidium along with potassium, lithium, and sodium. The lines of potassium and rubidium were photographed, but the photographic plates were not sufficiently sensitive to the lithium red line; it was therefore, as a rule, observed by the eye only; that is to say, spectroscopically, but not spectrographically. But whenever the rubidium line or lines were photographed, the red lithium line was invariably visible with the spectroscope. Thus, in sixty-two iron ores, rubidium was found in sixty-one. In sixteen red hæmatites, which are among the purest minerals found in the massive state, rubidium was detected in three. In seven magnetites, rubidium was contained in four. In seventeen aluminous minerals containing only very minute traces of potassium and sodium, rubidium was not detected. All the 170 minerals described contained copper, and of these 153 contained silver likewise. Lithium therefore was associated with copper, and likewise with silver. The presence of lithium was invariably observed in the spectra of the Bessemer flame, the number of observations certainly exceeding 100. Facts of the second kind relate more particularly to the salts of the alkalis; the crude material from whatever source invariably contains lithium; and many potassium salts, whether re-crystallised or otherwise, contain lithium and rubidium, and, not rarely, cæsium also.

Facts of the third category are derived from experimental evidence which is quantitative and spectrographic. With Wratten and Wainwright's plates (pinachrome bathed), which I have used for the last three years, it is possible to photograph far into the red, so that not only the potassium line λ 7667, but also the brownish red pair of rubidium lines with wave-lengths 7950·46 (Lehmann) and 7799 (Ramage) may be photographed with ease.

For some years past I have been engaged in determining the spectra obtained by operating on definite quantities of different salts, and am in a position to state the minimum weight of certain of the elements contained in each ore or mineral analysed, a weighed quantity of 0·5 grm. having been taken for each analysis.

The following passages refer to numbers taken from a communication read on June 16th, and since published by the Royal Dublin Society:—

Lithium.—When there are four lines visible on the photograph, namely, λλ 7608, 4603·07, 4132·93, and 3232·82, there cannot be less than 0·0089 grm. of lithium present in the flame. When the lines λλ 6708·0 and 4603·07 are visible, the quantity is not less or more than 0·041 grm. of lithium. When only the red line appears, the quantity is not more than 0·002 grm., and with half this quantity the red line ceases to be photographed.

Hence the quantity present, even when the red line is not photographed, may be as much as 0·001 grm. in 0·5 grm. of substance, or 2 parts of lithium in 1000; that is to say, 0·2 per cent. When care is taken to separate the alkalis this proportion is rendered much smaller.

The photographs were very generally taken upon Edwards's isochromatic plates, or those of Cadett and Neale; consequently the quantitative results with lithium and potassium are not comparable with those obtained more recently with Wratten and Wainwright's panchromatic plates. For instance, the most persistent line on the lithium plates was λ 4603 in the blue; now it is the red line λ 6708 which still appears strong after the blue line has ceased to be photographed; the spectrographic reaction therefore is now much more delicate.

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

† It may be stated here that a marine boiler incrustation deposited from sea-water was found by spectroscopic analysis to contain calcium and strontium, the latter in the proportion of about 1 per cent. But neither Mr. Ramage nor I, who both examined this material independently, could detect the presence of any lithium, the explanation being that it had remained dissolved in the water in the form of sulphate.

Potassium.—The ultimate line, that is to say the most persistent one in this spectrum, is λ 4047·39, and this is not photographed with less than 0·0000004 grm. of the element; this, when operating on 0·5 grm., indicates the presence of 0·00008 per cent of potassium. The flame reaction for potassium is therefore seen to be about forty thousand times more sensitive than that for lithium.

Rubidium.—Similar results were obtained with rubidium salts. All potassium salts not specially prepared as already stated were found to contain this element. The two rubidium lines 4215·8 and 4202·4 almost coincide with two iron lines, 4216·3 and 4202·1. When much iron is present it should be separated, but in instances where the penultimate line is visible, but the ultimate line is obscured by an iron line, this is not necessary because we know that this latter line must be there by reason of its being the stronger of the two rubidium lines. When these two lines are visible but very faint, there cannot be more than 0·00001 grm. of rubidium present. Smaller quantities are detected by the visibility of the ultimate line with wavelength 4202·4.

The connection between rubidium and lithium is this: that in whatever substance the less refrangible of the rubidium lines was photographed, lithium was detected by eye observations made on the flame with the spectroscope.

The question whether lithium is likely to occur or does occur in dust, may be answered by giving a list of substances in which it has actually been found.

It was found in mother-liquors from which potash-alum had been crystallised. In Donegal kyanite, which contains 98 per cent of aluminium silicate; in the quartz, felspar, and mica of granite from the Dublin and Wicklow mountains; in asbestos; in barytes from Duneen Bay, Clonakilty; in kaolin from Devonshire; in ordinary clay tobacco-pipes; in clay from decomposed quartzite found at Shankhill, Co. Dublin; in bauxite from France, and from Co. Antrim; in beryls from Glencullen, Co. Wicklow; and in many and various limestones.

In the Bessemer Flame.—At Crewe works of the London and North-Western Railway Company; at Middlesbrough in the Cleveland district of Yorkshire, and in South Wales.

In Atmospheric Dust.—In dust which was precipitated in sleet, hail, snow, and rain respectively, which fell in Dublin. It was not found in a specimen of meteoric dust which fell from the clouds, but it has been detected in some specimens of stoney meteorites, and in volcanic dust.

In Flue Dust.—In was found in flue dust from Crewe gasworks; Boyd's Chemical Works, Dublin; Nicholson's Copper Smelting Works, Leeds; ferro-manganese furnaces, Pittsburg, U.S.A.; ferro-manganese furnaces in England; Cleveland blast-furnaces; basic iron furnaces; and in South Wales iron furnaces; in the ashes of plants; in coal-ashes; also in soot from domestic chimneys. In short, twenty-four specimens of dust all contained lithium, and in nineteen of these rubidium was also detected. It was found in roasted shale and in smaltine. It occurs likewise in the gault of Cambridgeshire, as was ascertained by the late Sir George Stokes. Metallic calcium always contains it, and I have found it in a specimen of metallic palladium prepared by Wollaston the discoverer of this metal. It may be mentioned here that a careful examination of photographs taken of the oxyhydrogen flame spectra of flue dust from copper works enabled Mr. Ramage and myself to identify the following elements and determine their presence:—Sodium, potassium, lithium, rubidium, cæsium, copper, silver, calcium, strontium, aluminium, gallium, indium, thallium, iron, nickel, cobalt, manganese, chromium, lead. zinc, cadmium, and tin. Metals of the alkalis are not unfrequent constituents of heavy metals obtained by smelting processes; potassium occurs, for instance, in iron and steel; in the latter metal it was proved to be more than a mere surface contamination by immersing a new file in hydrochloric acid so as to remove any traces of adhering dust, washing thoroughly in pure distilled water, and burning the file in the oxyhydrogen flame.

One or two practical points occur to me to be worth mentioning. The spectrograph has a train of four quartz prisms of 60°, each one being composed of two halves cut from crystals of right and left-handed rotation; the collimator and camera lenses are also of opposite rotation, and have a focal length of about 12 inches for the D line; the dispersion and fine definition are such that with minute quantities of sodium the two yellow lines appear on the photographs so well separated that the distance from one to the other measures 0·42 mm. with a difference in wavelength of 6 Angström units.

Secondly, the supports used are pure ashless filter-papers 15 cm. in diameter, ten of which show nothing of a metallic nature when burnt but the sodium lines and possibly a trace of calcium.

It is not usual to take more than one of these filter-papers, and that is burnt in two halves; they are protected from any possible contamination through dust settling on them. All reagents used were prepared for the purpose, and blank experiments were made with them; moreover, all chemical reactions were carried out in platinum vessels.

I am therefore unable to corroborate the statement that sodium and potassium are elements much more widely distributed than lithium, or that lithium is an unlikely constituent of dust, glass, or solutions of copper, because all evidence obtained from flame spectra at high temperatures leads entirely to a contrary conclusion.

Moreover, there does not appear to be any obvious or particular connection between lithium and the radio-active elements, seeing that the former is "one of the most widely distributed elements in nature," and that, beside the alkali metals, we must place the following metallic elements:—Calcium, copper, silver, lead, manganese, aluminium, gallium, and iron.

COLLOIDAL CHEMISTRY.*

By H. R. PROCTER, M.Sc.

MODERN colloidal chemistry may be said to begin with the work of Graham about 1861, though the colloids had long been known to chemists, and Faraday (*Phil. Trans.*, 1857, i., 145) had prepared and described colloidal gold solutions. Graham first pointed out the radical differences between the colloid and crystalloid state, and introduced most of the nomenclature now in use, such as "sol" and "gel" for the apparent solution and the precipitate or jelly, and distinguished their various liquid media by the prefixes "hydro-," "alco-," &c. It would be impossible to refer in detail to the various workers who have since contributed to its advance, but the following may be mentioned as marking distinct steps in its progress. Wiedemann (*Verh. d. Physik. Ges. zu Berlin*, 1884, p. 44) showed that the absorption of water by solid colloids is in its earlier stages accompanied by considerable evolution of heat, actual solution and the fusion of jellies by absorption, while in gelatinisation heat is evolved. He later published researches on electrical osmose and cataphoresis which have important bearings on colloidal theory. The work of Van Bemmelen, 1888 *et seq.*, on colloidal jellies and precipitates is of permanent value, and his theory of the network or cellular structure of jellies has been very generally accepted by later workers, though, in your reporter's opinion, on insufficient evidence. In 1889 M. Carey Lea published extensive researches on "allotropic" (colloidal) silver (*Silliman's Journ.*), and in 1892 Picton and Sinder's work on "Solution and Pseudo-solution" (CHEMICAL NEWS and *Journ. Chem. Soc.*) opened new ideas on the nature of colloidal solution, and perhaps of solution in general. In 1893 Siedentopf and Zsigmondy,

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

building on the earlier work of Tyndall on the scattering and polarisation of light by non-homogeneous media, invented the ultramicroscope, in which, by a special form of dark-ground illumination, particles which were much too small to give an actual microscopic image were rendered visible as points of light. By this means the suspended matter in many colloid solutions has been rendered visible and the number and size of the colloid particles estimated.

Since sodium and potassium chlorides have been obtained in colloidal form in organic solvents, it is no longer possible to distinguish between colloid and crystalloid *substances*, but only between the colloid and crystalloid *state*. In the typical crystalloid solution of an electrolyte the dissolved body is separated into its molecules, and to a large extent into individual ions, while in the colloid sol the units of distribution are either large and often conjugated molecules or more frequently minute particles composed of many molecules united by cohesive attraction. Whether there is in principle any clear line dividing these conditions, or whether they do not rather shade off into each other by insensible gradations, is very doubtful, but in typical cases the distinction is sufficiently marked. As osmotic pressure and all its attendant effects are proportional to the *number* of the dissolved molecules, and therefore for a given quantity of substance inversely to the mass of the molecules or particles, these physical effects are almost absent in the colloid sol, and in most cases diffusion, lowering of freezing-point, raising of boiling-point, and *a fortiori* electric conductivity which demands ionic dissociation, are barely, if at all, perceptible, and may perhaps be attributable to the traces of electrolyte which appear essential at least to inorganic colloid solution. In the cases of many organic colloids of known high molecular weight, boiling- and freezing-point determinations have yielded numbers not inconsistent with the view that the particles are actual single molecules. While crystalloid bodies can usually only be separated from solution by evaporation or freezing of the solvent, or by chemical changes, colloid sols are unstable, and liable, either spontaneously or through very slight disturbing causes—such as the addition of an electrolyte, change of temperature, or concentration—to separate into an amorphous precipitate, or "gel," and the practically pure solvent. While crystalloid solutions are optically homogeneous, all colloid sols exhibit, in a greater or less degree, the "Tyndall effect" of scattering and polarising light, and, in most, minute suspended particles may be detected by the ultramicroscope. The relation of colloid sols to mechanical suspensions of very finely divided solids and to emulsions of immiscible liquids is a very close one, and may depend merely on the size of the particles. Many sols are visibly opalescent, but those with very small particles may be perfectly transparent, and metallic sols are often deeply coloured; gold sols vary from deep red to bluish violet.

The organic colloid sols differ in many respects from the inorganic, often approaching very closely to true solutions; and being usually bodies of high molecular weight and little subject to electrolytic dissociation, their true solutions would necessarily possess many of the properties associated with colloids. It is in fact by no means impossible that very large molecules or molecular aggregates may disperse and polarise light, and may even be made visible by the ultra-microscope. Organic sols are usually more stable than inorganic, and are flocculated or precipitated by different causes, so that it will be appropriate to defer their consideration till after that of the simpler and better investigated inorganic, or, as they are sometimes called, "suspension" colloids.

Inorganic colloid sols are invariably suspensions of very finely divided substances in media in which they are extremely insoluble. Von Weimarn ("Z. Chem. u. Ind. d. Kolloide," 1907, ii., p. 76) has shown that even the most definitely crystalloid bodies may be obtained in colloidal or gelatinised solution if produced in solutions in which they are extremely insoluble. An ordinary precipitate, such as barium sulphate, is at first formed by the combination of the ions in very minute crystal elements which are sufficiently soluble in water to rapidly unite to form crystals, which gradually increase in size by absorbing the smaller crystal elements. It is well known in ordinary analytical work that such precipitates will pass at first through any filter, but gradually become filterable by the effect of time and warmth. If, however, their insolubility is sufficiently increased by large excess of a common ion, or by effecting the combination in a medium, such as methyl alcohol, in which sulphates are extremely insoluble, the molecules cannot coalesce in the crystalline form, and a colloidal sol results. Even sodium and potassium chlorides have been obtained in colloidal form by the double decomposition of their organic compounds in organic media in which the salts were sufficiently insoluble. Though inorganic sols are precipitated by the addition of an electrolyte, the presence of free dissociated ions in small quantity seems essential to their formation and existence. If a solution of ferric chloride be dialysed through a colloid membrane into water, the hydrochloric acid formed by hydrolysis passes out into the water, leaving the ferric oxide in the dialyser as a colloid sol, and this goes on till the sol is practically free from chlorine, but if absolutely the last traces are removed, the ferric oxide flocculates and precipitates. Conversely, many oxide or hydroxide sols may be obtained by washing the precipitated hydroxide sufficiently free from electrolytes, or the precipitated and washed hydroxide may be re-dissolved by boiling with a very small quantity of neutral salt or free acid, when the precipitate will gradually pass into colloidal solution. This process (peptisation, Anätzung) has been extended to the colloidal solution of mechanically finely powdered oxides by the agency of small quantities of acids or salts, and has been employed by Kuzel (*D. R. P. Ammeld*, 12*g*. *K*. 30900 of Dec. 12, 1905) to bring many metallic oxides into the plastic condition requisite for the formation of the so-called "colloid" electric lamp filaments, now rapidly superseding the carbon fibre. Colloid sols are frequently formed by the free dilution of salts liable to hydrolytic dissociation, and especially salts of weak organic acids which from their low electrolytic dissociation do not readily cause precipitation. Even ferric chloride at a dilution of 1 : 125000 is completely dissociated in the cold in twenty-four hours, and much stronger solutions suffer partial dissociation, especially on heating. This is the cause of the darkened colour of hot ferric solutions. Acetates are still more readily hydrolysed, and especially by boiling, by which the free acetic acid is volatilised. Many solutions of hydroxides in alkalis are colloidal, but those in ammonia are usually true complex salts. Colloidal solutions are obtained of sulphides of metals of which the oxides, like those of arsenic and antimony, are soluble in water, by precipitation with H_2S, only water and the sulphide being formed in the reaction; and metallic sols of gold, silver, platinum, and other noble metals are formed by reduction of their dilute solutions, especially with organic reducing agents. Many sols may also be made by precipitation in presence of organic "protective colloids" which prevent precipitation by electrolytes. Thus silver halides may be obtained in colloidal solution in presence of gelatin, a fact of great importance in photographic plate making. An interesting point is that in many cases the nature of a sol may be changed by oxidadation, reduction, or chemical substitution without destroying its colloidal character.

An important method of producing metallic sols is that of Bredig (*Zeit. Angew. Chem.*, 1898, 951; *Zeit. Elektr.*, 1898, iv., 514), which differs in many respects from those just described. He employs the metal as cathode of an electric arc formed under water, which forms a sol with the particles projected from the cathode. Svedberg (*Ber.*, 1905, xxxviii., 3616) has applied the same method to the production of sols in ether, isobutyl alcohol, and other organic liquids, and of carbon, silicon, sulphur, and phosphorus in water (*Ber.*, 1906, xxxix., 1713). Bredig and Haber (*Ber.*, 1898, xxxi., 1741) have also shown that

under certain conditions sols can be produced by electrolysis, especially in alkaline solution.

The ultra-microscopes of Siedentopf and Zsigmondy ("Zur Erkenntniss der Kolloide," Jena, 1905) has rendered many details of colloid sols accessible to direct observation. In its original and most complete form it consists of a rectangular cell on the stage of the microscope, through the side of which a powerful horizontal beam of light is concentrated by appropriate lenses and adjustable slots; but several other types have been constructed for special purposes. No light can reach the observer but that from the brilliantly illuminated particles, which are thus rendered visible irrespective of size, as stars are visible to the naked eye on which the most powerful telescope can raise no measurable disc. As the field of view and the vertical thickness of the beam can be accurately measured, it is possible to count the visible particles in a given volume of liquid, and as the total concentration is known, their weight (and on certain assumptions as to form, their size), can be calculated. While the diameter of the smallest particles which could be seen by ordinary illumination, even with a magnifying power of 2250 diameters, is about 140 $\mu\mu$ (mm. $\times 10^{-6}$), Zsigmondy has observed particles in gold sols of from 20 to 80 $\mu\mu$ (submicrons), while still smaller particles (amicrons) exist, which scatter and polarise light, but cannot be separately distinguished. Zsigmondy estimates the size of the smaller of these amicrons as from 1·7 to 3 $\mu\mu$ (0·0000017 to 0·000003 mm.). Ostwald (Grundriss, p. 82) gives the probable size of a hydrogen molecule of molecular weight 2 as about 0·16 $\mu\mu$. As organic substances certainly exist having a molecular weight of several thousand, it seems quite probable that some of them may be visible to the ultra-microscope. Vanim and Hartl (*Ber.*, 1906, xxxix., 1696) have shown that if a small quantity of a ready-formed gold sol be added to a solution of a gold salt and a reducing agent, the colloid particles act as nuclei for the reducing gold. It is thus possible to build up amicrons to sub-microns which can be seen and counted. Some similar action takes place in gold ruby glass, which has been shown by Zsigmondy to owe its colour to colloid gold particles, but which is sometimes colourless until, by reheating, the gold germs have been allowed to grow by absorbing amicrons or reducing gold salts.

Other metallic and inorganic sols appear to contain particles of the same order of magnitude as the gold sols, but different sols vary considerably, and any sol contains a mixture of particles of various sizes.

As seen by the ultra-microscope, the particles are not at rest, but in constant rapid vibrating movement, and, according to Zsigmondy, gold particles have also a translatory motion. This vibration, which certainly plays its part in the physics of colloid sols, is identical with the Brownian or pedetic motion common to all minute particles suspended in liquid, but more marked in the ultra-microscopic, since it increases inversely with the size of the particles. This motion increases with temperature and diminishes with increased viscosity of the liquid, but is permanent and unaffected by time, or apparently by the exclusion of radiant heat and light. All theories therefore which ascribe the energy involved to outside influences would seem to be excluded, but it must be remarked that it is impossible to *observe* the phenomenon without light, and, ultra-microscopically, without light of a very intense character, so that the view suggested by Regnauld (*Journ. d. Pharm.*, [3], 1857, xxxiv., 141), that it was due to the warming of the particles by radiant heat, cannot perhaps even yet be entirely ignored. Sir W. Ramsay (Chemical News, 1892, lxv., 90) suggested that the motion was directly due to the heat-vibration of the water-molecules, though he thought it necessary to assume highly complex water-molecules to account for the disturbance of the much heavier solid particles. Einstein (*Ann. Phys.*, [4], 1905, xvii., 549, and 1906, xix., 2) and Smoluchowski (*Ann. Phys.* [4], 1906, xvii., 756) have discussed the phenomenon mathematically from two entirely different physical standpoints, and have reached results which not only show a scarcely expected concordance with each other, but a close relation to observed facts. The experimental work of Svedberg (*Ark. för Kemi, Min. och Geol.*, 1907, ii., *Nr.* 34) also strongly supports this view, so that, without presuming to discuss the mathematical evidence, it appears that a strong case is made out for regarding the motion as a direct consequence and evidence of the kinetic theory of heat. It may be remarked that if external sources of energy can be excluded, heat-motion seems the only available cause, since all the energy involved in overcoming internal friction of the liquid is converted into diffused heat, which in a space of uniform temperature cannot be re-transformed into any other form of energy.

If an electric current of sufficient strength be passed through an aqueous electrolysable solution in which the anode and cathode are separated by a diaphragm of porous earthenware, the liquid will pass through the latter towards the cathode till a certain equilibrium pressure is established. The phenomenon is known as "electrical osmose" or "cataphoresis." Although observed by Reuss in 1809, the subject was first systematically investigated by Wiedemann (*Ann. Phys.*, [2], 1852, lxxxvii., 321; and "Die Lehre der Elektrizität," 1893, Bd. I., 993). He showed that, with uniform difference of pressure, the quantity of liquid which passed through the diaphragm was proportional to the current-strength and independent of the area or thickness of the porous plate; and the pressure attained, when equilibrium was reached, to the potential difference between the two sides of the diaphragm. Quincke (*Ann. Phys.*, 2], 1861, cxiii., 513), substituting a capillary tube for the diaphragm, confimed these laws, and showed further that the equilibrium-pressure increased with diminished bore and relatively increased surface of the capillary; and that not only the pressure, but the direction of the flow were dependent on the nature of the material of the capillary and the liquid employed; oil of turpentine flowing to the anode in a glass capillary, and in the reverse direction in one lined with sulphur. It is obvious, therefore, that the action is between the surface of the liquid carrying the electric current and that of the capillary, and that if the liquid were fixed and the capillary surface free to move, it would be carried in the opposite direction to the flow of the liquid. This will be the case with suspended particles; and it has long been observed that these are carried in one or other direction according to their material and that of the liquid, most substances moving toward the anode in water and the cathode in turpentine, with a velocity proportional to the current-strength, but independent of their distance from each other and of the electromotive force. Quincke assumed that the particles possessed electrical charges different from the liquid, and usually negative as compared to aqueous solutions; so that in a current the − particle would tend to move to a region of higher + potential; that is, towards the anode. The relation of these facts to the contact-theory and to the phenomena of frictional electricity is obvious; a stream of liquid forced through a capillary of different potential would necessarily give rise to an electric current if furnished with appropriate conductors.

Helmholtz supported and somewhat amplified the hypothesis of Quincke (*Ann. Phys.*, [3], 1879, vii., 337). According to him, the limiting surfaces of two bodies of opposite electric character form a "double layer" (Doppelschicht); the + charge of the one layer exactly equalling the − charge of the other, and thus exerting no outside effect. Under the influence of a fall of potential the liquid layer tends to move over the other, and by friction communicates its motion to the adjacent liquid. Billitzer (*Wien. Ber.*, 1903, cxii., 95) supposes that the liquid coating of the double layer communicates its charge by diffusion to the remainder of the liquid, and, as this charge is constantly renewed by contact-action, both liquid and particles assume permanent charges, so that the latter behave, in a certain sense, like free ions. It is possible, or

perhaps probable, that the particles owe their original charges to the actual attachment of free ions by their residual affinities. It is not necessary to suppose, or even probable, that when two or more atoms unite to form a molecule their affinities are quantitatively saturated, but merely that, under the conditions, the residual affinity is insufficient to attach another atom. Under these circumstances it is likely that a mass of molecules may be able to hold additional ions in a way probably identical with what is known as "adsorption"; and these ions will affect the charge of the mass. The bearing of this on the phenomena of flocculation will be referred to later.

(To be continued).

DYNAMIC ISOMERISM.*

A.

Since the appearance of the last Report the experiments on the influence of acids, bases, and salts in accelerating isomeric change in solutions of nitrocamphor have been completed and published (Lowry and Magson, "The Influence of Nitrocamphor on the Mutarotation of Nitrocamphor," *Trans. Chem Soc.*, 1908, xciii., 107).

B.—Carbonyl Chloride as an Agent for Arresting Isomeric Change.

The most important feature of the year's work has been the discovery of a group of agents by means of which the isomeric change of nitrocamphor can be retarded or completely arrested. As long ago as 1899 (Lowry, *Trans. Chem. Soc.*, 1899, lxxv., 211) it was observed that solutions of nitrocamphor in chloroform behaved in an abnormal manner in that isomeric change was sometimes arrested during a period of one or two weeks and then abruptly started by some slight change of conditions, *e.g.*, by transferring the solution from a flask to a polarimeter tube.

The abnormal properties of this solvent were again noticed in the recent experiments on the influence of acids and bases. The basic chloroform solutions showed a marked decrease of activity when kept for a few days, whilst dilution produced a loss of catalytic power out of all proportion to the decreased concentration of the base. In one extreme case the isomeric change in a solution to which a trace of piperidine had been added practically ceased at the end of a fortnight, although the final condition of equilibrium had not by any means been reached. The more concentrated acid solutions produced an acceleration of isomeric change, but a retardation was noticed when smaller concentrations were used; in one case the rotatory power of the solution remained absolutely constant during a period of twenty-four days.

The earliest observations of arrested isomeric change had led to the conclusion that in non-oxygenated solvents the change was conditioned by the presence of a third substance, probably a basic impurity. The retardation produced by the acid chloroform solutions could not be attributed to mere neutralisation, since an acceleration was observed when benzene was used as the solvent. It was noticed, however, that the chloroform solutions acquired a pungent and unpleasant odour, which was ultimately traced to the formation of carbonyl chloride by oxidation of the chloroform $CHCl_3+O=COCl_2+HCl$; to this substance the curious behaviour of the chloroform solutions was undoubtedly due.

The action of the chloride probably depends on its power of converting ammonia and bases such as piperidine into neutral carbamides—

$$COCl_2+2NH_3=2HCl+CO(NH_2)_2,$$
$$COCl_2+2NC_5H_{11}=2HCl+CO(NC_5H_{10})_2.$$

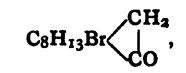
It was found that the chloride was capable of retarding or arresting the isomeric change of nitrocamphor dissolved in benzene or in ether, and that acetyl chloride produced a similar effect in benzene and in carbon disulphide. Phenylcarbamide did not retard the isomeric change of nitrocamphor dissolved in benzene, and acetyl chloride proved inefficient when added to a solution in acetic acid. In the latter case it is probable that the solvent has itself a catalytic action, and this may also be true when water and alcohol are used. The bearing of these observations on the problem of chemical change has been discussed elsewhere (*Trans. Chem. Soc.*, 1908, xciii., 119).

C.—Relationship between Absorption Spectra and Isomeric Change.

As the result of a series of experiments on the absorption spectra of substances of the ethyl aceto-acetate group, Baly and Desch (*Ibid.*, 1904, lxxxv., 1029; 1905, lxxxvii., 766) were led to the conclusion that there was a close relationship between the absorption bands which appeared in the spectra of some of these compounds and the property which they possess of undergoing reversible isomeric change. In view of the importance of this hypothesis in relation to the subject of dynamic isomerism it was thought to be desirable to test it in the case of a number of optically active compounds. These have the advantage that many of them can be crystallised out in a pure enolic or in a pure ketonic form, that it is possible by means of polarimetric observations to determine the actual velocity with which they undergo isomeric change, and that in a number of cases the proportions in which the isomerides are in equilibrium can be determined by means of solubility measurements. Photographs have therefore been taken of the absorption spectra of the majority of the camphor-derivatives which have formed the subject of previous investigations, and we are now in a position to summarise the principal results that have been obtained.

Halogen-derivatives of Camphor.

1. The camphor band observed by Baly and by Hartley at a concentration of N/10 practically disappears when the concentration of the alcoholic solution is reduced to N/100 (thickness of liquid 250 to 2·5 mm.), but can be restored by the addition of alkali. β-Bromocamphor,—



at a concentration of N/100 behaves in the same way as camphor.

2. α-Chlorocamphor and α-bromocamphor at a concentration of N/100 show a band in the same position $\left(\frac{1}{\lambda}\,3400\right)$ as the camphor band. The band is eliminated when the second α-position is occupied by bromine or by a nitro-group, but remains unaffected when the halogen is introduced in the β- or in the π-position.

3. No marked change in persistence is produced by the addition of alkali to α-chlorocamphor, α-bromocamphor, or their β- and π-bromo-derivatives. All these compounds are quite stable when alone, but undergo isomeric change rapidly in presence of a small proportion of alkali. It is therefore evident that in this case at least there is no direct quantitative relationship between the velocity of isomeric change and the persistence of the absorption-band.

Nitro-derivatives.

4. Nitrocamphane shows no absorption band, whether the solution be prepared by dissolving the crystals of the normal form or by adding acid to the sodium salt of the pseudo form. Even in presence of ten equivalents of alkali no definite band is developed. It is therefore clear that dynamic isomerism, even of a well-pronounced type, is not necessarily associated with the production of a band.

* Report of the Committee, consisting of Professor H. E. Armstrong (Chairman), Dr. T. M. Lowry (Secretary), Professor Sydney Young, Dr. C. H. Desch, Dr. J. J. Dobbie, Dr. M. O. Forster, and Dr. A. Lapworth. (Drawn up by the Secretary). Read before the British Association (Section B), Dublin Meeting, 1908.

5. Nitrocamphor dissolved in alcohol shows a shallow band at $\frac{1}{\lambda}$ 3000; it differs in position from that produced by bromocamphor—a result that is in accord with the view, consistently advocated, that the mobile hydrogen atom in this compound wanders to the nitro and not to the carbonyl group. The band disappears in the α-chloro and α-bromo derivatives, and also in the anhydride derived from the pseudo form by loss of water; its occurrence appears therefore to be limited by the same conditions as those which determine the occurrence of isomeric change.

6. In marked contrast with the behaviour of the —CHX.CO— compounds described above, the nitrocamphor band is enormously intensified by the addition of alkalis. This result is, perhaps, associated in some way with the fact that nitrocamphor forms a stable sodium salt whilst bromocamphor does not.

7. Camphoryl oxime—the isomer of nitrocamphor—resembles it in giving a very intense band in presence of alkalis, although the head of the band occurs at a considerably smaller wave-length $\left(\frac{1}{\lambda}\ 3650\right)$. The band does not appear in solutions of the oxime alone, and is not given by the acetyl derivative. Camphoric adhydride, the parent-substance from which the oxime is derived, is very diactinic, and shows no band.

Sulphonic-derivatives of Camphor.

8. β- and π-sulphonic derivatives containing the group—

$$Y.SO_2.C_8H_{13}\begin{cases}CHX\\ |\\ CO\end{cases}$$

usually show an absorption band, but this does not appear in camphor-β-sulphonamide and some of its derivatives. The replacement of both α-hydrogen atoms by halogens causes the band to disappear.

D.—Camphorcarboxylic Acid.

Investigations of the mutarotation phenomena and absorption spectra of camphorcarboxylic acid and its derivatives are in progress, but as the work is not yet complete it is proposed to reserve the discussion for a subsequent report.

THE FORMATION OF MISTS IN PRESENCE OF RADIUM EMANATION.

By Mdme. CURIE.

In a previous article I showed that the presence of radium emanation causes the condensation of saturant or non-saturant water vapour, as well as of other vapours. This condensation is manifested by the formation of a persistent mist, visible by the light of an electric arc (*Comptes Rendus*, December, 1907).

I have shown that the centres of condensation can neither be the ions formed in the gas nor the molecules of the emanation. It might be supposed that they are the particles of induced radio-activity suspended in the gas acting either by the electrostatic attraction of their charge, or by chemical affinity. It might also be thought that under the action of the emanation chemical compounds, capable of absorbing the vapour until drops are formed, arise in the gas. The second opinion is confirmed by experience, and experiments performed with water vapour are very conclusive on this point.

The experiments were carried out in sealed 150 cc. glass bulbs containing the liquid or solid to be investigated, and the gas charged with emanation (air, carbon dioxide, or hydrogen). To take an observation each bulb was placed in a water-bath and lighted by a convergent beam of light issuing from an electric arc. The quantity of emanation was approximately the same in the different experiments; it was the limiting quantity which can be emitted by 0·03 grm. of radium chloride. Blank experiments were performed with bulbs which did not contain the emanation. The mists due to the emanation are persistent, and can easily be distinguished from those which are produced by inequalities of temperature. They may last more than a month, and they disappear gradually in consequence of a gradual diminution in the number of drops as the emanation decays.

With pure water and air charged with emanation a persistent mist is observed; this mist is always slight, and it lasts some days. If the air is replaced by carbon dioxide no persistent mist is observed. But if, instead of using pure water, a mixture of equal weights of water and sulphuric acid is used, a dense mist, which is generally very fine at first, is obtained both in presence of air and of carbon dioxide; this mist can be observed for more than a month. The formation of mist is thus much facilitated by the addition of sulphuric acid to the water, when the pressure of the water vapour is greatly decreased.

In these experiments the particles of induced radio-activity cannot act directly as centres of condensation, for these particles exist in carbon dioxide as well as in air; moreover, their action would become more energetic with saturant than with non-saturant water vapour. It is natural to suppose that if we are dealing with water and air the centres of condensation are the nitrated compounds formed in air in presence of the emanation. If the mixture of sulphuric acid and water is used we are led to the supposition, though it may appear strange, that particles of sulphuric anhydride are formed in the gas owing to the action of the emanation on the liquid. A very persistent mist is also obtained in a flask which contains concentrated sulphuric acid and emanation in carbon dioxide; this mist lasts more than a month, while the much more intense mists which can be produced in another flask by local heating last less than a day.

Various experiments have been performed to demonstrate the *rôle* of chemical reactions.

If a flask which contains water and air charged with emanation is not sealed up but closed with a rubber cork, the mist obtained is much more dense and lasts much longer. It might be supposed that in this case the formation of centres of condensation results from chemical action on the cork, with oxidation of the organic matter and the sulphur. This supposition is confirmed by the following experiment:—A sealed glass bulb contains water, air charged with emanation, and a glass rod, on which is a piece of fused sulphur, and which is not immersed in the water. A fine mist appears in the flask and lasts for a month; after the experiment traces of sulphuric acid can be detected in the water.

Mists which are very intense at first and last more than a month have been observed with petroleum ether and carbon disulphide in presence of air charged with emanation; in the second case, the change in the liquid is very rapid, as shown by the formation of brown membranes. Anhydrous ether in presence of carbon dioxide and emanation also gives a persistent mist. In all cases probably the organic substances are attacked.

I have also examined certain solid substances, and I have obtained dense mists of long duration by using iodine in carbon dioxide charged with emanation, and camphor in air charged with emanation. On the other hand, if iodine is used with air and emanation, the mist only lasts for some days. During the dense mist visible deposits are produced on the walls of these flasks which do not contain liquid.

A mist may be observed with actinium in presence of water and carbon dioxide.

I have examined the action of an electric field on the mist formed in presence of the emanation. For this purpose I used a litre flask in which there were two parallel plates forming electrodes. The mist was obtained by means of a mixture of water and sulphuric acid; the volume being large it was necessary to use much more emanation than

before. I have proved that, as a whole, the drops behave as if they were not charged. It is true that they are violently attracted towards the electrodes, so that the mist rapidly disappears; nevertheless, this is only an effect of the displacement of uncharged particles in a field which is not uniform; the attraction acts towards the edges of the two electrodes, and the appearance of the displacement is independent of the sense of the field.

The formation of mist in a vessel which contains emanation may be regarded as an index of chemical reaction. Some experiments have been performed in my laboratory by M. Hirszfinkiel to determine the products of the reactions which may have occurred in the flasks used for my experiments. The most definite results were as follows:—Formation of a little carbon monoxide from the dioxide (a reaction already observed by Ramsay and Cameron), formation of nitrated compounds in the air, formation of traces of sulphuric and sulphurous acids in presence of sulphur and air.

M. Barkow has recently studied the condensation of water vapour under the influence of the Röntgen rays, of ultra-violet light, and of the penetrating radiation of radium. He frequently obtained condensation with slight expansion and attributed it to the formation in the gas of certain chemical compounds, such as nitrated compounds (*Ann. d. Physik.*, 1907).

The mists which I observe are composed of fine mobile drops, which are carried along by slow movements produced in the middle of the gas. When such a mist is not visible it often appears as if a much finer mist could be seen in the flasks which contain the emanation, although their existence could not be demonstrated with certainty.

The emanation must be fairly concentrated at first in order to produce the mist, which, however, may persist for a month when the concentration of the emanation has been reduced about 200 times.—*Comptes Rendus*, 1908, cxlvii., 379.

ACTION OF RADIUM EMANATION ON SOLUTIONS OF COPPER SALTS.

By Mdme. CURIE and Mdlle. GLEDITSCH.

A year ago Ramsay and Cameron announced in various publications that they had observed the production of alkali metals and of lithium in solutions of copper salts which had been subjected to the action emanation. They concluded that, in presence of the emanation, the metal copper undergoes a degradation into elements of the same family of lower atomic weight—potassium, sodium, lithium (*Nature*, July, 1907; *Journ. Chem. Soc.*, September, 1907; *Comptes Rendus*, 1908; *Archives de Genèvre*, April, 1908).

These important results attracted much attention, and it seemed desirable to reproduce them in laboratories which possess a sufficient amount of radium.

The experiment was performed as follows:—A solution of a copper salt (sulphate or nitrate) was placed in a little glass flask into which a large quantity of emanation was introduced and allowed to decay there spontaneously. The copper was then separated, the remaining solution evaporated to dryness, and the residue examined. The same operations were carried out with a solution of the same copper salt which had not been subjected to the action of the emanation. The experiments were repeated several times. The residue consisted chiefly of a sodium salt (with a little K and Ca); in the four experiments described, in which the emanation had been used, the presence of lithium was observed by means of a spectroscope. In the blank experiments the residue was much smaller, and no lithium could be detected. Ramsay and Cameron performed an experiment to determine the amount of lithium present; they found about 0·00017 mgrm. of lithium in a residue which weighed 1·67 mgrm. obtained from 0·27 grm. of copper (0·815 grm. of copper nitrate), while in the corresponding blank experiment the residue was only 0·79 mgrm. (This weight of metallic lithium does not correspond to the amount indicated by the mixture of sodium and lithium salts used for comparison, and there must have been an error which we have not been able to detect).

We tried to repeat the experiment, taking every possible precaution. The experiment is delicate, and is liable to be affected by many sources of error, the chief of which is the use of a glass vessel, as Ramsay himself has stated.

Our preliminary experiments have shown that it is extremely difficult to get chemical products free from lithium.

It is found in distilled water and in almost all reagents; if a reagent does not contain it, and is allowed to remain some time in a glass vessel, it is then found to contain some traces of the element. The following experiment was performed:—Water which had been distilled in a platinum still and kept in a platinum flask, left no visible residue when 250 cc. were evaporated in a platinum dish, and the last drop formed by the concentration process did not give the lithium spectrum. But if water obtained in the same way is kept in a glass flask for twenty-four hours, after evaporation a small residue is obtained, consisting chiefly of a sodium salt, but containing also a trace of lithium.

It seemed to us indispensable to replace the glass by some other substance. We have proved that it is equally dangerous to use quartz, a substance which was employed by Ramsay, for commercial quartz vessels contain lithium. We treated the fragments of a dish of opaque quartz and a piece of a transparent quartz tube with hydrofluoric acid free from lithium; a considerable amount of lithium could be detected in the residue. Transparent quartz contains much more than opaque quartz. We therefore decided to use platinum vessels.

The apparatus we used consisted of a cylindrical platinum receiver 7·5 cm. long and with an external diameter of 1·5 cm. placed horizontally; at the end of this receiver there was a little vertical platinum tube through which we introduced the solution. The little tube had a platinum lid, which protected the solution, but did not act as an air-tight stopper. A glass tube was soldered externally over the platinum tube; it was furnished with a side tube with a stopcock. The solution was introduced into the apparatus by means of a platinum syphon, and was withdrawn in the same way, and was never allowed to come in contact with the glass parts of the apparatus.

The water and the acids used for the experiment were re-distilled in a platinum still, and kept in platinum bottles. We found that all the reagents contained lithium, especially the sulphuric acid. After the purification process the presence of lithium could not be detected in the residue from the evaporation of 80 cc. of nitric acid, 25 cc. of sulphuric acid, 25 cc. of hydrofluoric acid, and 250 cc. of water.

As Ramsay had observed, commercial pure copper salts contain considerable amounts of lithium.

We tried various methods of purification—repeated precipitation with sulphuretted hydrogen, deposition of copper by electrolysis, fractional crystallisation; we finally used copper sulphate which had been subjected to many crystallisations in a platinum dish, pure water being always used for re-dissolving. At first this treatment is very efficacious, but it is very difficult, if not impossible, to remove the last traces of lithium. After the purification had been performed, it was possible with difficulty to detect lithium in the residue from the treatment of 50 grms. of copper, but none could be detected in the residue from 2 grms. of salt.

The emanation was obtained from a solution which contained 0·19 grm. of radium (0·25 grm. $RaCl_2$). It was first condensed in a worm immersed in liquid air, and then drawn into the apparatus. In order that the quantity of emanation used might be known exactly we measured the radiation emitted from the apparatus by comparison with that of a known amount of radium. For the measurement a specially constructed condenser with large plates was used.

Two experiments analogous in every respect were performed. About 7 cc. of a solution of pure copper sulphate were introduced into the apparatus; this liquid presented a large free surface relative to its volume. The emanation was introduced by degrees, and in order to make sure that it dissolved, the solution was shaken by inclining the apparatus, which was placed in melting ice. This process was repeated several times. The weights of metallic copper employed were 0·26 and 0·14 grm. The whole quantity of emanation introduced was in both cases measured by the saturated emanation of 0·37 grm. of radium; the quantity of emanation which was actually destroyed in the apparatus was rather less; it was measured by the saturated emanation of 0·27 grm. of radium. When the experiment was thought to be finished the solution was transferred from the apparatus into a platinum dish, and some drops of nitric acid were added. A platinum electrode was introduced into the same dish, and the copper was deposited on it. The solution after being freed from copper was evaporated to dryness in the dish, and heated until the sulphuric acid was driven off. The residue was dissolved in a few drops of water, and treated with sulphuretted hydrogen to remove the traces of copper still present. The liquid was filtered through a little platinum funnel on to a platinum lid of known weight, and evaporated to dryness at a very moderate temperature. The very small residue obtained was weighed.

Seven cc. of a solution of pure copper sulphate which had not been exposed to the action of the emanation was subjected to the same treatment. The residues finally obtained were examined with a spectroscope. Their weights were 0·0004 and 0·0005 grm. in the first experiments, and 0·0003 and 0·0002 grm. in the blank experiments.

It must be noted that the amount of copper used is about the same as that employed by Ramsay. The quantity of emanation used is also approximately the same (1·85 cmm. of emanation according to Ramsay's method of estimation). Nevertheless, the residue finally obtained is much smaller.

The spectroscopic examination showed that this residue contains principally sodium and a little potassium; the presence of lithium could not be proved. An experiment performed with mixtures of sodium and lithium sulphates showed that it is possible, though with difficulty, to detect the presence of the red lithium line with a mixture which contains 1 part of Li_2SO_4 in 10,000 parts of Na_2SO_4, and that it is easy to see the same line with a mixture containing 1 part of Li_2SO_4 in 3000 parts of Na_2SO_4. Consequently, the quantity of metallic lithium which could be present was less than $0·6 \times 10^{-5}$ mgrm.

With the same amounts of copper and of emanation, Ramsay and Cameron state that $1·7 \times 10^{-4}$ mgrm. of lithium was present. If, owing to a clerical error, this number represents lithium chloride, the amount of metallic lithium would be 3×10^{-5} mgrm.

The residue which we obtain is always much less than that obtained by Ramsay and Cameron, and this is probably due to the rejection of glass vessels. The difference in the weights of the residues obtained by us in the direct and in the blank experiments is very small (0·1 to 0·3 mgrm.); this is probably because in the experiment performed with the emanation the entrance of the latter might lead to the introduction of traces of foreign matter. In Ramsay and Cameron's most complete experiment this same difference was 0·88 mgrm., and we think that it might be attributed to the more energetic attack upon the glass by the solution in presence of the emanation.

The following control experiment was performed:—Into a solution of copper sulphate containing 0·27 grm. of copper we introduced the amount of lithium sulphate corresponding to $1·7 \times 10^{-4}$ mgrm. of LiCl; this solution was then treated in the same way as in the experiments described above. With the residue finally obtained it was easy to see the red lithium line, which proves that the lithium was not lost during the treatment.

Thus, we may say that we have not succeeded in confirming Ramsay and Cameron's experiments. It is evidently impossible to assert that no trace of sodium or lithium is formed in the experiment, but we consider that the formation of these elements cannot be regarded as proved.—*Comptes Rendus*, 1908, cxlvii., 345.

THE ELECTROLYTIC PROPERTIES OF DILUTE SOLUTIONS OF SULPHURIC ACID.*

By W. C. D. WHETHAM, M.A. F.R.S., and H. H. PAINE, B.A., Trinity College, Cambridge.

(Continued from p. 130).

PART II.—*By* H. H. PAINE (*continued*).

8. *The Conductivity Curve.*

WE are thus led to consider once more the cause of the drop in the equivalent conductivity curve. The evidence seems conclusive against the supposition that the presence of carbonic acid, or indeed of any other acid, is sufficient to explain the phenomenon.

In the same series of experiments as those with exhausted water, referred to in the first part of the paper, the effect of the presence of carbonic acid deliberately introduced into the solution was ascertained (*Roy. Soc. Proc.*, A, lxxvi., 580). If instead of plotting the k/m curve as was there done, we plot the total conductivity of the solution against the concentration of sulphuric acid—both for the solution containing carbonic acid and that made up from the pure distilled water—we shall find that the curve for the former case always lies above that for the latter, the two gradually merging as the concentration increases. The difference between the ordinates of the two curves at any particular concentration gives us the effect of the carbonic acid on the conductivity. The effect is always positive, but diminishes in magnitude as the concentration of the sulphuric acid increases. This is explained simply and logically on the supposition that the carbonic acid behaves as a "second acid," contributing its partial conductivity to the whole conductivity of the solution. Since it is a very weak acid, its ionisation is suppressed by the hydrogen ions of the sulphuric acid, and hence its effect will become less and less, ultimately becoming negligible, as the concentration of the sulphuric acid increases. Hence, by subtracting the full initial conductivity of the solvent containing carbonic acid, we make the partial conductivity of the sulphuric acid appear smaller than it really is, and thus apparently get a drop in the k/m curve. If the purest distilled water contained carbonic acid, the presence of this impurity would explain part of the drop observed, but there remains an excess effect to be accounted for in another way.

For let us consider the equivalent conductivity curve for a solution made up with the purest water obtained by distillation in air. If, instead of deducting the conductivity of the solvent, we take the total conductivity of the solution in plotting the k/m curve, we shall find that the curve still falls off at extreme dilutions. Since the effect of carbonic acid is always additive, correcting for it, to get the true conductivity of the sulphuric acid, can only exaggerate the drop in this curve. Hence on the basis of any known phenomenon, the presence of carbonic acid, or of any other free acid, is insufficient to explain the whole of the apparent diminution in the conductivity of the sulphuric acid.

Water distilled *in vacuo* has a much smaller conductivity than that distilled in air; hence most of the impurities in the latter are dissolved from the air. Now the atmospheric gases most likely to be dissolved in any appreciable quantity are carbonic acid and ammonia. Normally there is forty times as much carbonic acid as ammonia in the atmosphere, but the solubility of ammonia is so much greater than that of carbonic acid that the quantities of each which would be

* A Paper read before the Royal Society, June 18, 1908.

present in solution are probably of one and the same order.

Let us then regard the solvent as containing ammonium carbonate, which at such dilution would be largely in the ionised state. The addition of a strong acid will result in the formation of un-ionised carbonic acid—the ammonium ions remaining in solution. Some of the hydrogen ions of the strong acid have thus been removed. We may regard the first small quantity of acid added as being removed from the solution or rendered inactive, and the partial conductivity as due to the portion left.

Again, instead of adding an acid, let us introduce a strong alkali like potash. Here we shall have un-ionised ammonia (or ammonium hydrate) formed, and some of the hydroxyl ions of the strong alkali will be removed from the solution. As before, the effect is the same as though the first portion of alkali added had been nullified.

This view is best made evident from the conductivity curve—plotting the "partial conductivity" of the acid (the conductivity of the solution minus the initial conductivity of the solvent) against the mass of acid added. A quantitative as well as a qualitative agreement with facts may be observed. The curve is a straight line. When produced it does not pass through the origin, but cuts the axis of zero conductivity at a point showing the concentration of acid to be $4{\cdot}7\times10^{-6}$ grm.-equivalent per litre (for a curve plotted during the course of the experiments described in this paper). The effect on the conductivity therefore is the same as though this quantity of acid had been neutralised or removed from solution. Now from the migration constant experiments, since the rise in the measured migration constant was only half what we should expect if the whole of the conductivity of the solvent had been due to saline matter, we may conclude that about half this quantity of salt was present in the water, giving a conductivity of about 5×10^{-7}. Suppose this salt to be ammonium carbonate. The velocity of the CO_3 ion (deduced from the migration constants of potassium and sodium carbonates and sulphates) is about 50, while that of the (NH_4) ion (deduced in a similar manner from the figures for ammonium chloride, &c.) about 70. Hence $u+v=120$. We then get $k=m(u+v)=5\times10^{-7}$, so that $m=5\times10^{-7}/120=4{\cdot}2\times10^{-9}$ grm.-equivalent per cubic centimetre, or $4{\cdot}2\times10^{-6}$ grm.-equivalent per litre.

If this be the quantity of ammonium carbonate present in solution, it would account for the neutralisation of an equivalent quantity of sulphuric acid. The agreement with the number deduced from the conductivity curve is very close indeed.

It was also noticed that, when exposed to the air, the conductivities of dilute solutions of acid diminished, often at a considerable rate. That no solution of alkali from the glass produced this effect was shown by preventing the air from getting into contact with the solution, when the conductivity remained quite constant. This seems to show that ammonia, or some other alkali, is readily absorbed from the atmosphere—the hydrogen ions being replaced by ammonium ions in the solution, and the total conductivity being diminished.

There is an objection which must be faced. A solution of ammonium carbonate, on boiling, gives off ammonia and carbonic acid. Hence, in the exhaustion experiments described in the first part of this paper, when the conductivity of the solvent was considerably reduced before the addition of the sulphuric acid, we might have expected that some of the ammonium carbonate would have been abstracted, and the effect made apparent on the k/m curve. It must be remembered, however, that the last traces of ammonia would be removed only with great difficulty. Further, from the above calculations it would appear probable that half the conductivity of the solvent is due to the presence of something which does not affect the transport ratio. If this represents an excess of carbonic acid, the results are explained. Since ammonia is much more soluble than carbonic acid, the latter would be evolved much more readily on exhausting the water. Hence the diminution in conductivity would be accounted for by the removal of the carbonic acid, while the ammonia present may be almost unaffected.

9. We have sought to explain the apparent diminution in the equivalent conductivity of acids (and similarly of alkalis) at extreme dilution, by the association of ions brought together in solution. Such as association would take place if the solvent contained :—(1) A weak acid such as carbonic acid, (2) a weak alkali such as ammonia, or (3) a salt formed by the neutralisation of a weak acid by a weak alkali, such as ammonium carbonate. We have seen that experimental evidence is opposed to the supposition that the presence of carbonic acid alone is sufficient to explain the phenomenon ; we have concluded that the presence of ammonium carbonate is capable of providing this explanation. It has been shown that the diminution in the equivalent conductivity would be accounted for if by some means the conductivity of $4{\cdot}7\times10^{-6}$ grm.-equivalent of sulphuric acid per litre were destroyed. Hence the "impurity" in the water must be such as will produce this electrical effect. Now consider the three cases. In the first, we should get approximately the same result electrically, if there were 5×10^{-6} grm.-equivalent of ionised carbonic acid per litre of the solvent, which becomes un-ionised on the addition of the strong acid ; in the second, $3{\cdot}5\times10^{-6}$ grm.-equivalent of ionised ammonia per litre would be sufficient; and in the third, 5×10^{-6} grm.-equivalent per litre of ammonium carbonate—also in the ionised state. The conductivities of these solutions would be—

1. $k_1 = 5\times10^{-9}(319+50) = 1{\cdot}8\times10^{-6}$,
2. $k_2 = 3{\cdot}5\times10^{-9}(184+70) = 0{\cdot}9\times10^{-6}$,
3. $k_3 = 5\times10^{-9}\ (70+50) = 0{\cdot}6\times10^{-6}$.

The actual conductivity of the water was between $0{\cdot}9\times10^{-6}$ and $1{\cdot}0\times10^{-6}$. Hence there could not have been sufficient carbonic acid present in the free state in the distilled water to account for the observed "diminution" in the conductivity of the solution. The conductivity of the water easily allows for the presence of sufficient ammonium carbonate. The case for ammonia is just on the border-line. The high velocities of the hydrogen and hydroxyl ions make the number of grm.-equivalents of an acid or alkali required to produce a given conductivity, less than that of a salt. That is why, for a given conductivity, ammonium carbonate is more effective in reducing the conductivity of the solution of sulphuric acid than ammonia or carbonic acid present separately.

We have assumed throughout this final investigation that the velocities of the ions remain constant for the greatest dilutions. It is still possible that these velocities diminish, and that the above explanation only partially accounts for the drop in the k/m curve. To complete the solution of the problem, it would be necessary, either to obtain an exact knowledge of the impurities in the water, so as to allow for their effect, or to use water which contained no such impurities. In the water distilled in air, it is probable that ammonia and carbonic acid are dissolved ; if so, the effect in diminishing the conductivity necessarily follows.

The Staffordshire Society.—The Council of the above Society is making a special effort to increase the membership. It is well known there are many Staffordshire men in London and the provinces, most of whom would, no doubt, appreciate the objects of the Society, which are to promote social intercourse, cordiality, and good fellowship ; also to further objects of interest to the County. The Society offers excellent opportunities for bringing together friends from their own County. The Hon. Secretary of the Members' Committee, Mr. E. A. BARNETT, 2, Audley Road, Hendon, N.W., will be pleased to receive names and addresses of Staffordshire men who desire to become members of the Society.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 108).

The Iron Group (continued).

Procedure 65.—When phosphate is present, test one-tenth of the HNO_3 solution (P. 63) for iron, by evaporating it just to dryness, adding 1—2 cc. HCl (1·20), evaporating again to decompose the HNO_3, diluting to 5 or 10 cc. and adding 5 cc. KCNS solution. (Permanent red colour presence of *iron*). To the remainder of the solution add NH_4OH (0·96) until the precipitate formed by the last drop does not re-dissolve on shaking. If, owing to the addition of too much NH_4OH the solution becomes alkaline or a large precipitate separates, make it distinctly acid with acetic acid. Add 5 cc. of a 50 per cent solution of ammonium acetate, and, unless the mixture is already of a brownish red colour, add 10 per cent $FeCl_3$ solution drop by drop until such a colour is produced. Add enough water to make the volume about 100 cc., boil in a 250 cc. flask for five minutes, adding more water if a very large precipitate separates, and let the mixture stand for a minute or two. Filter while still hot, and wash with hot water. Add 3—5 cc. more ammonium acetate solution to the filtrate, boil it again, and collect on a separate filter any further precipitate. Make the filtrate alkaline with NH_4OH, adding an excess of 2—3 cc., filter off and wash any precipitate, uniting it with the main precipitate. (Filtrate, P. 66; precipitate, reject or dissolve in hot HCl (1·12), and treat by *P. 65*a*).

Notes.

1. With regard to the test for iron with KCNS and the necessity of removing the HNO_3, see P. 64, N. 2.

2. This method of separation depends on the facts that, upon boiling an acetic acid solution containing much acetate, ferric iron and titanium are completely precipitated, and thallic thallium and zirconium nearly so, in the form of a basic acetate or hydroxide; and that all the phosphate present combines with these elements when they are present in excess, and therefore it then passes completely into the precipitate, leaving the bivalent elements in solution. This behaviour of the phosphate is due to the fact that the solubility in acids of the phosphates of the trivalent and quadrivalent elements is much smaller than that of the phosphates of the bivalent elements.

3. If upon adding the ammonium acetate the solution becomes of a reddish colour, it shows that iron is present in quantity more than sufficient to combine with the phosphate; for a cold solution containing ferric acetate is of a deep red colour. If, on the other hand, a colourless solution results (either with or without a precipitate), it shows that there is no excess of iron, and $FeCl_3$ is therefore added, which causes the precipitation of $FePO_4$ as a yellowish white precipitate. Upon boiling, the excess of iron separates completely as a dark red gelatinous precipitate of basic ferric acetate, leaving the supernatant liquid colourless, except when nickel or cobalt is present.

4. The solution is diluted to at least 100 cc., owing to the large volume of the precipitate, and it must be heated in a capacious flask owing to its tendency to boil over.

*5. Zirconium may not be completely precipitated under the conditions of this procedure, 1 to 5 mgrms. sometimes remaining in solution, especially when considerable acetic acid is present. To ensure its complete separation, the filtrate is made alkaline with NH_4OH.

*6. The precipitation of thallium in this procedure is not quite complete, but there is no danger of losing even 0·5 of a mgrm. when iron is also present, as is always the case.

**Procedure* 65*a*.—To the HCl solution (P. 64 or 65, and *62*a*) which should be of specific gravity of 1·11—1·12, add 10 to 20 cc. more HCl (1·12), transfer the (cold) solution to a separating funnel, add an equal volume of ether, shake vigorously several times (preferably after inverting the funnel and opening the cock), and then allow the two layers to separate. Draw off the layers separately, and rinse out the funnel with a little ether. Return the aqueous layer to the funnel, and treat it with ether as before; if necessary, repeat this treatment once or twice until the ether layer remains colourless. (Water solution, *P. 65*b*; first ether solution, *P. 65*d*; remaining ether solutions, reject).

Notes.

1. If the directions are followed, 97 to 99 per cent of the $FeCl_3$ present passes into the ether layer in each extraction. It is evident from this statement, that even when 500 mgrms. of iron are present, substantially all of it will be removed in three extractions. But it is important that the concentration of the HCl solution in contact with the ether layer lie within the narrow limits of 20 to 22 per cent HCl, corresponding to a specific gravity of 1·10 to 1·11 at 15°. This concentration is realised in the procedure, even though a little stronger acid is used, for some of the HCl passes into the ether layer. The extraction of the $FeCl_3$ is less complete both with stronger and weaker HCl solutions; thus with HCl containing initially either 18 or 25 per cent about 94 per cent of the $FeCl_3$ passes into the ether layer; while with 8 per cent HCl only 4 or 5 per cent of the $FeCl_3$ was extracted in each shaking. Almost all the thallium, which is present as $TlCl_3$, also passes into the first ether extract.

2. The following are probably the principles involved in this ether extraction. Since iron passes into the ether layer only in the form of $FeCl_3$, the quantity of it extracted by the ether increases in accordance with the distribution law, the larger the proportion of un-ionised anhydrous $FeCl_3$ in the water layer. This proportion is, however, increased by increasing the concentration of HCl both in virtue of the reduction of the ionisation by the common-ion effect, and of the reduction of the hydrolysis of the ferric salt by the free acid. It is doubtless true that the strong acid has also a dehydrating effect, thereby increasing the anhydrous $FeCl_3$ in the water layer. As the HCl becomes very concentrated, however, another effect, opposite in character, comes into play; namely, ether dissolves in large quantities in the aqueous layer, and HCl and water dissolve in large quantity in the ether layer, thus making the two layers more nearly alike, and doubtless decreasing the distribution-ratio for the $FeCl_3$ between the ether and water layers. With respect to this explanation, it should be added that it is uncertain to what extent complex acids $HFeCl_4$, &c.) may be involved.

3. Since the colour of the ether layer is a sensitive indication of iron, the treatment with ether may be discontinued as soon as a nearly colourless ether extract is obtained. When titanium is present the water layer may remain distinctly yellow, owing to the presence of hydrogen peroxide as impurity in the ether; such a colour does not therefore show that iron is still present.

4. Titanium and zirconium remain completely in the water layer. When much zirconium is present some of it may be precipitated out as chloride, but this remains suspended in the water layer.

5. Phosphoric acid does not interfere with this separa-

† From the *Journal of the American Chemical Society*, xxx., No. 4.

tion, and the iron can therefore be extracted even after the basic acetate procedure.

Procedure 65*b*.—Heat the HCl solution (*P. 65*a*) on a water-bath until the ether is expelled, add 1 cc. H_2SO_4 (1·20), evaporate almost to dryness until the H_2SO_4 begins to fume, adding 1 cc. more H_2SO_4 (1·20) if the residue is solid. If the residue is dark coloured, owing to organic matter, add a few drops HNO_3 (1·42), and evaporate again until the H_2SO_4 begins to fume. Cool, add 5 cc. water, 10 cc. 3 per cent H_2O_2 solution, and then 10 cc. 10 per cent $Na_2HPO_4.12H_2O$ solution. (Orange-yellow to orange-red solution, presence of *titanium*; white flocculent precipitate, presence of *zirconium*). Let the mixture stand for at least an hour, filter, and wash the precipitate. (Filtrate, if coloured, *P. 65*c*; precipitate, Note 3).

Notes.

1, By the addition of H_2O_2 titanium is converted into a sulphate corresponding to the higher oxide TiO_3, the red colour being due to the cation. This colour test is an extremely delicate one, even 0·1 mgrm. Ti imparting a distinct yellow colour to the solution.

2. By the Na_2HPO_4, zirconium is precipitated as a basic phosphate, $Zr(OH)PO_4$. Its precipitation is slow; but, if nothing has separated after half-an-hour, it is safe to conclude that less than 0·5 zirconium is present, provided care has been taken not to use more H_2SO_4 than is directed in the procedure. If the titanium had not been oxidised by the addition of H_2O_2, it would also give an entirely similar precipitate of $Ti(OH)PO_4$; but from a solution containing H_2O_2 in excess when titanium is alone present none of it separates even on standing several hours. When zirconium and titanium are present together, a small proportion of the titanium is carried down with the zirconium, and the phosphate precipitate may then have a distinct yellow colour.

3. Some of the rare earth elements, such as thorium, may also be precipitated as phosphate at this point, if these elements have not been proved absent or removed in *P. 52*a*. In such a case the presence of zirconium may be proved by pouring a portion of dilute HF several times through the filter (supported in a celluloid funnel or a platinum ring), evaporating with H_2SO_4 in a platinum dish, diluting, and adding NH_4OH. The rare earth phosphate would be left undissolved by the HF.

4. Besides titanium and zirconium, the solution from *P. 65*a* will sometimes contain uranium, manganese, and cobalt (carried down in the precipitates in P. 52, *P. 62*a*, and P. 64, respectively), but not in sufficient amount to interfere with the zirconium and titanium tests. The only other element of the aluminium and iron groups that gives a similar colour with H_2O_2 in acid solution is vanadium; but this, aside from the fact that it should not be present, would give no precipitate with Na_2HPO_4 (not even on the addition of Na_2SO_3 in *P. 65*c*).

(To be continued).

CORRESPONDENCE.

APPOINTMENT OF PUBLIC ANALYST FOR THE METROPOLITAN BOROUGH OF SHOREDITCH.

To the Editor of the Chemical News.

Sir,—I am directed to bring to your notice the following letter which has been addressed to the Town Clerk of the Metropolitan Borough of Shoreditch,—I am, &c.,

Richard B. Pilcher,
Registrar and Secretary.

The Town Clerk, Metropolitan Borough of Shoreditch.

Dear Sir,—The attention of the Institute of Chemistry has been directed to the terms of an announcement to the effect that the Council of the Metropolitan Borough of Shoreditch is about to appoint a Public Analyst in the place of the late Sir Thomas Stevenson.

The Council of the Institute, while feeling that they need not impress on the Borough Council the great importance of securing the services of a fully competent analyst, desire me to express their regret that the appointment should have been offered in terms which might suggest that it is to be given to the candidate making the lowest tender.

My Council venture to express the opinion that, in the case of an appointment of this kind, the qualifications for which are of a purely personal character, the system of inviting professional men to tender for an appointment has the effect of lowering the status of their profession, to the detriment of the public service. It precludes, and in this case has precluded, many analysts of repute from becoming candidates for the appointment, and therefore limits the selection, while it is not likely that the Borough Council will secure the services of the best man by entrusting the position to the practitioner who places the least value on his skill, experience, and professional position.

I am desired to request you kindly to bring this letter to the notice of the Borough Council when the appointment in question is under consideration.—I beg to remain, dear Sir, yours faithfully,

(Signed) Richard B. Pilcher,
Registrar and Secretary.

A similar communication has been addressed to the Town Clerk of the City of York.

COMPULSORY WORKING OF PATENTS.

To the Editor of the Chemical News.

Sir,—Needless anxiety has been caused to hundreds of patentees by the inaccurate statements which have appeared in the public press concerning the provision contained in the Patents and Designs Act, 1907, relative to the "working" of patents in this country, and I regret that I find the same inaccuracy in Prof. Kipping's Address to the Chemical Section at the recent meeting of the British Association, as reported in your issue of 11th September.

The sole ground connected with "working" on which any person may apply for the revocation of a patent is that "the patented article or process is manufactured or carried on exclusively or mainly outside the United Kingdom."

It is only when he is satisfied that the allegation to the above effect by the person making the application is correct that the Comptroller can call upon the patentee to show that the article or process is being manufactured or carried on to an adequate extent in the United Kingdom.

It follows that patentees whose patents are not being "worked" abroad have no reason to fear for the safety of their patents on this ground.

The inaccuracy in no way affects Prof. Kipping's able review of the position of chemical industry in this country, but it is desirable that inventors should not be misled as to their position.—I am, &c.,

A. G. Bloxam.

Birkbeck Bank Chambers, W.C.,
September 14, 1908.

Oxidation of Isoeugenol. Dehydrodiisoeugenol.—H. Cousin and H. Hérissey.—The oxidation of isoeugenol with iron perchloride gives a white crystalline substance, which appears to be dehydrodiisoeugenol, isomeric with dehydrodieugenol. The same compound is formed by the oxidising action upon isoeugenol of the ferment in mushrooms. The authors have prepared the dimethyl, diacetic, and dibenzoic ethers of dehydrodiisoeugenol.—*Comptes Rendus*, cxlvii., No. 4.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 4, July 27, 1908.

Gases Occluded in a Special Nickel Steel.—G. Belloc.—The gases occluded in a nickel steel containing 45 per cent of nickel and 0·15 per cent of carbon are carbon dioxide, carbon monoxide, hydrogen, and nitrogen. In the turnings of the steel the total volume of gas is three and a-half times the volume of the alloy. The t, dv/dt curve is practically parallel to the temperature axis; the proportion of carbon monoxide increases up to 75 per cent, while the hydrogen has a maximum at 540° and then regularly decreases. With the wire the total volume of gas is ten times that of the metal; the t, dv/dt curve has two marked maxima—one at 540° and the other at 830°; the variations in the composition are not progressive. At 540° the carbon monoxide is at a minimum and the hydrogen at a maximum. Above this temperature the carbon monoxide increases irregularly. The alloy belongs to the class called by Guillaume "reversible," and at 350—400° the beginning or end (according as the temperature is increasing or decreasing) of the progressive transformation occurs. The extraction of most of the gases is effected when the iron is in the γ-state and the nickel in the β-state, and a regular increase in the proportion and composition of the gases such as occurs with the turnings is to be expected. The difference between the absolute volume of the gas extracted from the wire and the turnings may be explained by one of the following hypotheses:—(1) A very heterogeneous distribution of the gases in the alloy; (2) a loss of occluded gas due to the mechanical action of the drill; (3) an absorption of gas during the operation of drawing into wire.

New Method of Determining the Fixed and Volatile Acids in Wines.—Emm. Pozzi-Escot.—100 cc. of the wine are evaporated to 3 to 4 cc., treated with excess of alcohol, and filtered. The filter is ignited in a muffle furnace, and thus the insoluble salts of the wine are determined. Their alkalinity is determined by titration with nitric acid. Another 100 cc. of the wine are evaporated to 3 to 4 cc., neutralised with baryta, and 100 cc. of alcohol at 96°, containing 2 per cent of barium bromide, are added. The fixed acids are thus precipitated as barium salts, and the volatile acids remain in solution. The precipitate is filtered off, ignited, and titrated with nitric acid. The difference between the first titration and the second gives the fixed acids, A_f. The fixed acids minus the total acidity of the wine, A_t, gives the volatile acids, A_v. Thus, $A_f - A_t = A_v$. This method does not give exact results in presence of lactic acid, which, however, is exceptional in wines, and, moreover, it can only be used for organic acidity.

New Method of Preparing Mixed Anhydrides of Organic Acids.—J. Bougault.—When phenylisocrotonic acid is treated with iodine in presence of a very large excess of sodium carbonate, benzoylacrylic acid, $C_6H_5—CO—CH=CH—CO_2H$, is formed. If a sufficient quantity of the sodium salt of an aromatic acid is added to the aqueous solution containing sodium phenylisocrotonate and carbonate in excess, and then iodine is added gradually, the mixed anhydride of benzoylacrylic acid and the aromatic acid employed is obtained. By this method the author has prepared various mixed anhydrides, *e.g.*, benzoic-benzoylacrylic, $C_6H_5—CO—CH=CH—CO>O$, $C_6H_5—CO$, and has investigated their properties.

Constitution of Vicianine.—Gabriel Bertrand and G. Weisweiller.—Vicianine (a glucoside found in the seeds of *Vicia augustifolia*) resembles amygdaline, but is distinguished from it by its physical constants. It contains a CH_2O group more than amygdaline. With concentrated sulphuric acid in the cold a carmine red coloration is produced which is characteristic of the group $C_6H_5—CH<^{CN}_{O}$. The presence of this group is confirmed by two reactions:—(1) The liberation by hydrolysis with diastase of one molecule of benzoic aldehyde and one molecule of hydrocyanic acid. (2) The production of phenylglycolic acid by the hydrating action of mineral reagents. Thus vicianine, like amygdaline, is a derivative of lævophenylglycolic nitrile.

MISCELLANEOUS.

East London College.—The Drapers' Company have voted special grants for the purchase of important additions to the equipment of the Engineering Department, the staff and students of which have carried out a series of tests of vanadium and other special steels which are being largely used in the construction of high-speed engines. The recent installation in the Electrical Engineering Department of an 11 kilowatt rotatory converter of special design should be of great use to senior students, and to those seeking to carry out standardisation experiments. The list of academic distinctions gained by students of the College affords proof of the many-sided activity of a modern University College. In the Faculty of Arts, two students obtained the degree of Master of Arts in mathematics. In the Faculty of Science, one student is made Doctor of Science, and another Bachelor of Science for research in branches of Chemistry, a subject which is particularly well developed at the College. So far as the Internal Honours Examinations of London University are concerned the College does well in Science (three first classes), and almost as well in Engineering, where two first classes are won, although the Faculty of Engineering is here, as elsewhere, in point of numbers much smaller than either the Faculty of Arts or of Science. Mr. W. Lower Carter, M.A., has been apppointed Lecturer in Geology.

Birkbeck College.—The 86th Session of the College will commence on Monday, September 28th. An Opening Address will be given in the theatre, at 7.30 p.m., by Dr. Albert Griffiths. The College will be open for inspection after the Address. Visitors are invited. The College is conducted in close relation with the University of London; twenty-eight Members of the Staff are Recognised Teachers of the University; and the courses of study provide for Degrees in the faculties of Arts, Science, Laws, and Economics. The Laboratories are well-equipped with modern apparatus and appliances, and Research Work is encouraged in the various Science Departments (Chemistry, Physics, Botany, Zoology, and Geology). According to the Calendar more than one hundred Students passed some Examination of the University during the last Session; several Students gained Scholarships at the older Universities; one obtained the London University Scholarship in English, and one obtained a City Parochial Foundation Scholarship. Eleven Students gained Assistant Examinerships in the Patent Office, twelve gained appointments as Assistant Surveyors of Taxes, and other public appointments were obtained. The Arts Subjects include Courses in English, Classics, French, German, Italian, History, Logic, Mathematics, Economics, and Geography, all conducted by Recognised Teachers of the University. The Law Courses comprise Mercantile and Common Law, Equity, Conveyancing, and Patent Law, and Classes in English and Roman Law, and Constitutional Law and History. There is also an Art School, which provides instruction in the various Branches of Artistic Work (Painting, Modelling, Life, &c.). Classes are held both in the Day and in the Evening.

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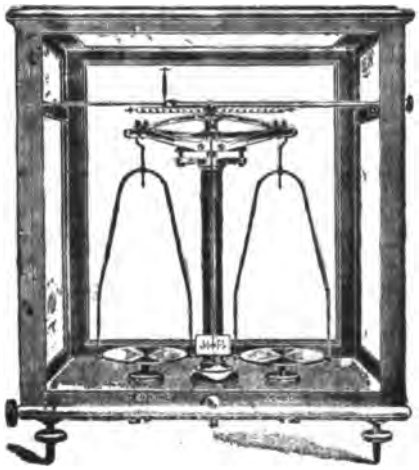

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A DEMONSTRATION IN THE RAPID ELECTRO-ANALYTICAL SEPARATION OF METALS.*

By HENRY J. S. SAND.

In the process of electro-analysis to be shown the principle of very vigorous stirring of the electrolyte has been combined with that of keeping the potential of the cathode under control by means of an auxiliary electrode. It has thus been possible very largely to extend the scope of electro-analytical methods. The following metals have hitherto been studied :—First, the metals of the silver and copper groups and zinc, *i.e.*, silver, mercury, copper, bismuth, lead, cadmium, and zinc. These metals have all been deposited singly, separated from each other, and also separated when all present in the same solution. In the last-named case the quantity of each metal taken varied between 0·10 and 0·15 grm., and the time for the deposition of each between about ten and fifteen minutes (*Trans. Chem. Soc.*, 1907, xci., 373). Secondly, new methods have been elaborated for the determination and separation from each other of antimony and tin. A considerable number of determinations have been carried out in which the metals taken in varying ratios weighed together approximately 1 grm. The time for the deposition of the antimony was usually about twenty minutes, that for the tin about eighty minutes. Lastly, the possibility has been examined of securing a purely electro-analytical method for the analysis of an alloy consisting of copper, antimony, lead, and tin, and all the separations required for such an analysis have at the present time been carried out.

THE PRODUCTION OF AMMONIA FROM ATMOSPHERIC NITROGEN BY MEANS OF PEAT.*

By H. C. WOLTERECK, Ph.D.

Attempts to utilise the nitrogen contents of peat have been made during the last sixty years, but the yield of ammonia obtained by the various processes which have been tried shows that only about one-third of the nitrogen can be thus recovered. Starting from an observation that a mixture of nitrogen and hydrogen passed over reduced iron at a low heat always produced ammonia for some time, the author made further experiments on these lines, and found that the presence of oxygen and water was of importance. This led to the gradual reduction of the quantity of hydrogen, and finally to its complete omission; but the periodical necessity of reducing the iron used for the purposes of moist oxidation led him to employ some carbonaceous material (coke, coal, charcoal, &c.), for this purpose.

Finally, peat was found to give the most satisfactory and rapid results, and even when it contained up to 80 per cent of water it could be utilised advantageously.

In the meantime experiments were made to prove the co-operation of atmospheric nitrogen. Sugar carbon absolutely free from nitrogen was treated by the process, and produced an average of 1 per cent ammonia on the carbon consumed.

The large plant erected at Carnlough, Co. Antrim, has finally confirmed the working and results of the process on a commercial scale.

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

THE INFLUENCE OF COLLOIDS AND COLLOID SUSPENSIONS ON THE SOLUBILITY OF CARBON DIOXIDE IN WATER.*

(Preliminary Note).

By ALEX. FINDLAY.

The problem of the absorption of gases, more especially oxygen and carbon dioxide, by blood is one which has exercised the minds of physiologists and physicists for many years, the view generally held being that the gases are retained in the blood largely owing to the formation of chemical compounds. With regard to the absorption of CO_2, the reputed alkalinity of the blood, and the consequent formation of carbonate and bicarbonate, have been held chiefly responsible for the increase in solubility of CO_2 in blood as compared with that in a corresponding salt solution. Recently, however, various investigators (Holber, Farkas, Fraenkel, &c.) have shown that blood is "water neutral," in which case the generally held view regarding the absorbtion of CO_2 by blood becomes untenable.

Believing that the colloids present in blood might very probably play an importan *rôle*, it was decided to undertake an extended investigation of the influence of colloids on the absorption of gases by water, since only a very few experiments in this direction have been carried out (Geffchen).

The following results have been obtained by Mr. W. H. Harby, B.Sc., working under the direction of the author. All determinations were made at 25°, and the solubility values represent the ratio of gas volume absorbed to the volume of the absorbing liquid :—

I. Solubility in water, 0·823. Experimental error about ± 0·25 per cent.				
II. Colloidal ferric hydroxide—				
Grms. of $Fe(OH)_3$ in 100 cc. of liquid	1·36	0·68	0·34	0·136
Solubility of CO_2 .	0·896	0·877	0·837	0·824
III. Gelatin—				
Grms. of gelatin in 100 cc. of liquid .	5	3	1	
Solubility of CO_2 .	0·848	0·834	0·826	
IV. Arsenious sulphide—				
Grms. As_2S_3 in 100 cc. of liquid .	0·305	0·244	0·122	0·0244
Solubility of CO_2 .	0·822	0·825	0·825	0·825
V. Silicic acid—				
Grms. H_2SiO_3 in 100 cc. of liquid .	0·113		0·068	
Solubility of CO_2 .	0·823		0·822	
VI. Dextrin—				
Grms. of dextrin in 100 cc. of liquid .	20	10	5	1
Solubility of CO_2 .	0·735	0·757	0·768	0·817
VII. Soluble starch—				
Grms. of starch in 100 cc. of liquid .	10	5	1	
Solubility of CO_2 .	0·776	0·801	0·815	
VIII. Glycogen—				
Grms. of glycogen in 100 cc. of liquid	1·18		0·263	
Solubility of CO_2 .	0·804		0·820	
IX. Albumen—				
Grms. of albumen in 100 cc. of liquid	0·5		0·75	
Solubility of CO_2 .	0·822		0·822	

Experiments, still in progress, have been made with the white of egg, diluted with five times its volume of water. An increase of about 8 per cent in the solubility of CO_2 was found. Soon, however, a precipitate began to form, and the solubility of CO_2 diminished, some of the gas which had been dissolved passing out of solution. Finally, there was an increase in the solubility of about 4 per cent.

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

THE TRANSFORMATION OF AROMATIC NITROAMINES AND ALLIED SUBSTANCES, AND ITS RELATION TO SUBSTITUTION IN BENZENE DERIVATIVES.*

I. *Transformation of Nitroaminobenzenes into Nitroanilines.*

(With W. W. Reed, M.Sc., A.I.C.

The investigation of the transformation of 2·4-dichloro-1-nitroaminobenzene into 2 : 4-dichloro-6-nitroaniline, reported on last year (*Brit. Assoc. Reports*, 1907, p. 101) has been continued.

Transformation of the Crystalline Nitroamine.

The object of the new experiments on the transformation of the crystalline nitroamine was to determine with greater accuracy the limiting value of the pressure of the gaseous catalyst, below which no transformation occurred. The method employed was substantially that described in last year's Report, but more elaborate methods were used for determining the partial pressure of the reagent, hydrogen chloride. The form of the experimental tube was so modified that, after the change had occurred (or failed to occur), the tube could be opened, attached to a Sprengel pump, and its exact volume measured by collecting the air, filling it at a given temperature and pressure. The hydrogen chloride was introduced as dilute standardised solutions, known weights of which were used. A series of such experiments has shown that at 13° the hydrogen chloride must be at a pressure of 14 mm. of mercury for a change to take place.

Transformation of the Nitroamine in Solution.

Inasmuch as the nitroamine is colourless, whilst the nitroaniline is intensely coloured, the change can be quantitatively followed by comparing the colour of the solution, during the course of the transformation, with that of standard solutions of the nitroaniline. The method is exceedingly delicate; with practice it is easy to detect the difference in tint between 49 and 50 cc. of a solution of nitroaniline containing 0·000225 grm. in 100 cc. when the tint of these volumes is compared in columns 12 cm. in length; that is 0·00225 mgrm. of the aniline can be measured.

The low solubility of the nitroaniline (100 cc. at 14° dissolve 0·0022 grm.) allows only of the use of solutions of the nitroamine containing 0·003 grm. per 100 cc. in these experiments.

The measurements of the speed of transformation show that the reaction is one of the first order. The change into the nitroaniline is accompanied by a conversion (reduction) of the nitroamine into diazonium salt to the extent of 30 per cent. This fact only permits of the determination of an apparent value of the velocity coefficient.

Solvent.—The only solvents which could be used were water, acetic acid, or mixtures of these. Alcohol and acetone, which are solvents of the catalysts, react with the nitroamine.

As the proportion of the acetic acid in the solvent is increased the rate of change slowly rises until, with 74 per cent acetic acid, the rate is double that in pure water. But the use of higher concentrations of acetic acid is followed by a rapid rise in the speed of the change, which in glacial acetic acid is several hundred times as great as in water.

ii. *Nature of the Acid (Catalyst).*—The efficacy of acids as catalysts in this transformation is roughly in the order of their activities as measured by other reactions. Nevertheless, chloric and perchloric acids appear to bring about the change twice as rapidly as hydrochloric acid, although they are somewhat less active as catalytic accelerators in hydrolyses, &c. The reduction of the nitroamine to diazo-compound is, however, much less in the case of these acids, a fact which may account for the more rapid formation of nitroaniline.

Hydrobromic and hydriodic acids reduce the nitroamine; the former reacts quantitatively according to the equation $Ar.NH.NO_2 + 3HBr = Ar.N_2Br_3 + 2H_2O$, the diazonium perbromide crystallising out when acetic acid is the solvent. Hydriodic acid carries the reduction further.

iii. *Concentration of the Acid.*—The effect of the concentration of the acid catalyst on the rate of change has been carefully tested with several acids both in aqueous and acetic acid solution.

In aqueous solution the rate is proportional to the square of the concentration of the acid—that is, on doubling the concentration of the acid the velocity is quadrupled.

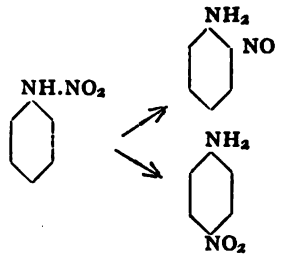
In acetic acid solution this proportionality does not hold, the increase of velocity being less for given increases of concentration of acid. For example, the ratio of the velocity coefficients for N/10 and N/20 H_2SO_4 in glacial acetic acid at 25° is $k_{N/10} : k_{N/20} = 2·14$ instead of 4.

iv. *Effect of Temperature.*—Withing a range of temperature of 0—35° the products of the transformation of the nitroamine were not modified. When the nitroamine was melted under water or other solvents, a more profound decomposition was observed accompanied by oxidation of the benzene nucleus.

In aqueous solution the rate of the transformation was greatly increased by a rise of temperature. At 25° the rate of change is approximately thirty times as great as at 0°; the ratio of the velocity coefficients, when 2N HCl is the catalyst, at 25° and 35° C. is $k_{35°} : k_{25°} = 0·002488/0·00108 = 2·3$.

Experiments on Nitroaminobenzene, $C_6H_5.NH.NO_2$.

A number of preliminary experiments have been made with the unsubstituted nitroaminobenzene. In its main features the molecular re-arrangement of this nitroamine resembles that of its dichloro-derivative, but it is complicated by the simultaneous formation of both *o*- and *p*-nitroaniline—



Experiment has shown that the latter is only formed to the extent of some 10 per cent, but the difference in the intensity of the colours of the solutions of the nitroanilines has so far made any accurate measurement of the rate of change impossible by the method above described. Under like conditions (temperature and concentration of catalyst) the velocity is about one and a-half times greater in the case of the unsubstituted than with the dichloro-compound; in glacial acetic acid, however, the change was roughly ten times as rapid.

Reduction to the diazo-compound also takes place, and since in this case the diazo-compound is unstable, the subsequent decomposition introduces another factor, which prevents quantitative measurements of the velocity.

The views expressed in last year's report are confirmed by these more extended observations. The most simple interpretation of the facts seem to be that the catalyst (the acid) and the nitroamine form an additive compound, which alone is capable of molecular re-arrangement.

For what reason the speed of the transformation should be proportional to the second power of the concentration

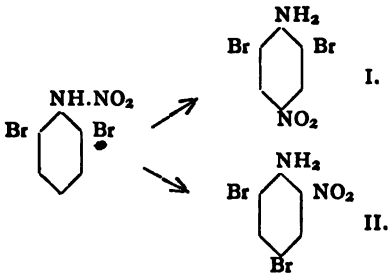
* Report of the Committee, consisting of Professor F. S. Kipping (Chairman), Professor K. J. P. Orton (Secretary), Dr. S. Ruhemann, Dr. A. Lapworth, and Dr. J. T. Hewitt. Read before the British Association (Section B), Dublin Meeting, 1908.

of the acid is not very obvious. Blanksma (*Recueil des Trav. Chim.*, 1903, xxii., 290) observed that the rate of the analogous transformation of acetylchloroaminobenzene into *p*-chloroacetanilide bore the same relation to the concentration of the hydrogen chloride, which is the only catalytic accelerator of this change. Acree (*Am. Chem. Journ.*, 1907, xxxviii., 258) has suggested that the existence of the additive compound of the chloroamine and the catalyst is conditioned by the equilibrium, $Ar.NClAc,HCl \rightleftarrows Ar.NCl.Ac + H^{\circ} + Cl'$, when the concentration of the complex would depend on the square of the concentration of the acid. That the ratios of the accelerating effects of hydrochloric, nitric, and sulphuric acids, $k_{Hce} : k_{HNO_3} : k_{H_2SO_4} = l : l : 2/3$, are approximately in their ratio of their ionisation in normal solutions is in harmony with this view.

II. *The Wandering of Bromine in the Transformation of Nitroaminobromobenzenes.*

(With C. Pearson, B.Sc.).

In the transformation of 2 : 6-dibromo-1-nitroaminobenzene, in all circumstances when such a change will take place, we have observed a remarkable re-arrangement of the points of attachment of the substituting groups.



Not only is the normal product, 2 : 6-dibromo-4-nitroaniline I., in which the nitro group passes to the vacant para-position formed, but also 2 : 4-dibromo-6-nitroaniline II. In the production of the latter compound, the migrating nitro group displaces one ortho-placed bromine atom, which then becomes attached to the para-carbon atom.

The direct nitration of 2 : 6-dibromoacetanilide is not accompanied by any such re-arrangement; the nitro group only enters the para-position.

NOTE ON A VOLATILE COMPOUND OF COBALT WITH CARBON MONOXIDE.*

By Dr. LUDWIG MOND, Dr. HEINRICH HIRTZ, and M. DALTON COWAP.

In a previous paper (*Journ. Chem. Soc.*, 1890, p. 749), published by one of us in conjunction with Dr. Carl Langer and Dr. F. Quincke, entitled "Action of Carbon Monoxide on Nickel," p. 752, we stated that "numerous experiments made to obtain similar compounds of carbon monoxide with other metals, notably with cobalt, iron, copper, and platinum, have only led to negative results." In a later paper (*Journ. Chem. Soc.*, 1891, p. 604), published by one of us in conjunction with Dr. F. Quincke, we announced the formation of iron carbonyls, which were subsequently more fully described by one of us in conjunction with Dr. Carl Langer (*Journ. Chem. Soc.*, 1891, p. 1091).

In continuing the experiments with cobalt and taking advantage of the method of Sir James Dewar for facilitating the formation of nickel carbonyl by acting upon the finely divided metals by carbon monoxide under a high pressure and correspondingly high temperature instead of working at ordinary pressure and ordinary temperature as we had hitherto done, we succeeded in obtaining small quantities of a cobalt carbonyl when the pressure was raised to 50 atmospheres and the temperature to 150° C., and the quantity formed was considerably increased by raising the pressure to 100 atmospheres and the temperature to 200° C.

The cobalt oxide was prepared from a pure oxalate, free from nickel and iron, by heating to about 200° C. The sketch exhibited gives the apparatus in which we have obtained the cobalt carbonyl. The cobalt oxide was filled into a steel retort, which was fitted with two steel pipes for the gas circulation. All iron parts were lined with copper so as to prevent the action of carbon monoxide on the iron. This retort was heated in an oil-bath. Behind the outlet valve we fixed a small gun-metal cylinder, which contained a filter of flock asbestos, and behind this we had an additional filter of cotton-wool in a glass tube. From there the gases had to pass a condensing bottle, and the spent gases were burnt at outlet. The cobalt oxide was placed into the retort and reduced with hydrogen at 5—10 atmospheres pressure, and at the lowest possible temperature. A slow current of carbon monoxide was then passed through the apparatus at 100 atmospheres pressure. The temperature of reaction lies between 150° C. and 200° C. Both filters were kept at 50° C. by means of warm water, The condensing bottle was cooled in ice. In this bottle we obtained large orange crystals of the cobalt carbonyl.

The analysis of these crystals was made by decomposing them with bromine water. The carbon monoxide was measured and the cobalt estimated in the solution, The result was :—

Co.	CO.	Co : CO.
0·0744 grm.	0·1597 grm.	1 : 3·96
0·0582 „	0·1115 „	1 : 4·03

We conclude, therefore, that this compound is $Co(CO)_4$, corresponding to the liquid nickel carbonyl.

The cobalt carbonyl is gradually decomposed in air, leaving a deep violet substance, which has not yet been investigated. The crystals are best preserved by sealing them in a glass tube under hydrogen or carbon monoxide. The cobalt carbonyl is very slowly attacked by non-oxidising acids, as hydrochloric or sulphuric. The reaction is accelerated by the presence of an oxidising agent. The carbonyl is quickly, almost instantaneously, decomposed by oxidising acids, such as bromine, chlorine, nitric acid, and the corresponding cobalt salt is formed under elimination of carbon monoxide, according to the following equation—$Co(CO)_4 + Br_2 = CoBr_2 + 4CO$. The cobalt carbonyl is insoluble in water, but more or less soluble in the several organic solvents, such as carbon bisulphide, ether, naphtha, alcohol, and nickel carbonyl. If these solutions are kept standing for some time, or if they are warmed, decomposition sets in. These and other solutions and their products of decomposition have not yet been investigated.

The specific gravity measured by the suspension method at 18° C. is 1·827, but we do not yet regard this figure as final, as the substance is so easily decomposed, and if only kept for a few hours the specific gravity increases considerably, due to decomposition. The vapour tension *in vacuo* could not be measured with certainty owing to decomposition. The vapour tension at 15° C. measured 11 mm., but already at 25° C., decomposition could be proved, which was already very marked at 35° C., so that we are not sure if there was not already a slight decomposition at 15° C. In a carbonic oxide atmosphere the decomposition commences at about 40° C. to 45° C., and is complete at 130° C. to 135° C., leaving metallic cobalt.

The melting-point lies between 42° C. and 46° C. We could not estimate it more exactly owing to its decomposition.

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

The authors reserve for themselves the right to further examine this reaction with other metals.

The Laboratory,
20, Avenue Road, Regent's Park, London, N.W.,
June 22, 1908.

SOME REACTIONS OF DICHLORO UREA.*

By F. D. CHATTAWAY, D.Sc., F.R.S.

UREA is so well known and has been so much investigated that any new simple substance obtainable from it posesses quite an unusual amount of interest. Such a new substance is found in dichloro urea, which, leaving out of consideration the derivatives of ammonia itself, is one of the simplest possible compounds containing halogen attached to nitrogen. It is produced when chlorine is passed into a cooled saturated aqueous solution of urea. Action takes place without any considerable development of heat, and dichloro urea crystallises out as a white powder consisting of small transparent plates. It can be preserved for a considerable time in a dry atmosphere, although it is not a very stable body. It is comparatively safe to handle, and promises to be of considerable use as a synthetic agent, as it is easily soluble in water, alcohol, and ether, and is very reactive. Its composition shows that it has the formula $CON_2H_2Cl_2$, and having regard to its mode of formation as well as to the structure of urea itself, its constitution is most probably represented by the formula $OC{<}^{NHCl}_{NHCl}$.

By means of this formula its formation and such of its reactions as have yet been studied can be explained. The other theoretically possible chloro substitution derivatives of urea, though probably formed in the reaction between chlorine and urea, have not so far been isolated.

When dichloro urea is produced, although two molecules of hydrogen chloride are formed, very little heat is developed, and it must therefore be an endothermic compound, and might be expected to be highly explosive. When heated, however, it does not itself explode, but decomposes at about 83°, with liberation of nitrogen chloride, which latter may detonate with great violence if not allowed to escape, and if the temperature is raised a few degrees higher, as, for example, when dichloro urea is heated in a test-tube over a water-bath.

Dichloro urea gives all the characteristic reactions of a typical nitrogen chloride; for instance, it liberates iodine from hydriodic acid, chlorine from hydrochloric acid, and reacts with alcohol, forming ethyl hypochlorite, urea being in each case re-formed.

Dichloro urea is distinguished from most other substituted nitrogen chlorides by the readiness with which it is hydrolysed, nitrogen chloride, carbon dioxide, a little nitrogen, and ammonium chloride being formed. If the compound is dissolved in water or kept in a moist atmosphere, this hydrolysis takes place slowly at the ordinary temperature, and becomes very rapid at about 30° C.

It is probable that in this reaction a mono-substituted ammonia is first produced thus—

$$CO{<}^{NHCl}_{NHCl} + 2H_2O = CO{<}^{OH}_{OH} + 2NH_2Cl.$$

But if so, it apparently can only exist momentarily, as nitrogen chloride is at once liberated. The formation of the end products of the reaction can be explained by assuming that this monochloro ammonia immediately breaks up into ammonia and nitrogen chloride. $3NH_2Cl = 2NH_3 + NCl_3$, which then to some extent react in the ordinary way, forming nitrogen and hydrogen chloride, the latter at once combining with the free ammonia, and allowing the remaining nitrogen chloride to escape, as this does not react with ammonium chloride.

Both acids and alkalis accelerate the rate of hydrolysis, and further alter the nature of the end products by hindering or furthering the secondary reaction between the ammonia and the nitrogen chloride. In presence of dilute acids the ammonia is at once fixed, and the reaction between it and the nitrogen chloride, with its accompanying liberation of nitrogen, is prevented; all the chlorine contained in the dichloro urea is therefore liberated as nitrogen chloride. In presence of alkalis, on the other hand, the reaction between the ammonia and the nitrogen chloride goes on to completion since the hydrochloric acid formed in it is at once fixed; no nitrogen chloride therefore is set free since twice as much ammonia as is required to decompose it is present.

The reaction between dichloro urea and a solution of caustic potash is very energetic; nitrogen is liberated with effervescence, the excess of ammonia and the alkaline carbonate formed remaining dissolved in the liquid. The action, which is quantitative, is expressed by the equation—

$$3CO{<}^{NHCl}_{NHCl} + 12KOH =$$
$$= 3K_2CO_3 + 2NH_3 + 6KCl + 2N_2 + 6H_2O.$$

This behaviour of dichloro urea with alkalis affords an insight into what occurs when urea is decomposed either by an excess of alkaline hypochlorite or hypobromite. The course of this reaction has never hitherto been explained, although it has received an unusual amount of attention on account of its furnishing a ready method of estimating the quantity of urea present in a liquid. In this reaction the urea is without doubt converted into a chloro or bromo urea, which is at once hydrolysed in the manner above described. It is possible that in presence of excess of hypochlorite or hypobromite a tri- or tetra-substitution product may be produced, the formation and decomposition of which can be formulated easily, but this does not affect the essential character of the reaction, which is one of halogen substitution followed by hydrolysis of the substituted urea and interaction between the resulting compounds.

A reaction which indicates the use to which dichloro urea may be put in the synthesis of simple carbon and nitrogen rings is that between it and ammonia.

When ammonia in excess is added to an aqueous solution of dichloro urea, hydrolysis, accompanied by liberation of nitrogen and formation of carbonate, occurs, but in addition diurea, $CO{<}^{NH.NH}_{NH.NH}{>}CO$, is produced, and separates in considerable quantity as a sparingly soluble crystalline powder. This is the first direct synthesis of diurea from urea itself, the compound having been previously obtained from ethyl carbonate and hydrazine.

This adds another to the very few reactions known by which nitrogen atoms can be made to link up together, and further affords an exceedingly simple synthesis of hydrazine.

Diurea, when heated with excess of strong sulphuric acid to a little above 100° C., is easily hydrolysed, carbon dioxide escapes, and hydrazine sulphate is produced. This crystallises out perfectly pure in almost theoretical amount on cooling and adding a little water.

Dichloro urea can be prepared from urea and converted into diurea so easily, and the latter can be hydrolysed so quickly, that the reactions afford a synthesis of hydrazine more adapted to prepare a small quantity than any yet described. The operations involved are so simple and can be carried out so quickly, that they are also excellently suited for showing the synthesis of a hydrazine as a lecture experiment.

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

Action of Arsenic Trichloride on Nickel and on Arseno-nickels. — Em. Vigouroux. — The action of arsenic trichloride on nickel or its arsenides between 400° and 600° gives the compound NiAs, coloured red as when it is found in nature, while at higher temperatures the shining white compound Ni_3As_2 is formed. When these substances are heated in a current of hydrogen in presence of arsenic they are transformed into a greyish powder of formula $NiAs_2$.—*Comptes Rendus*, cxlvii., No. 8.

COLLOIDAL CHEMISTRY.*

By H. R. PROCTER, M.Sc.

(Continued from p. 130).

The phenomenon of electric cataphoresis was first studied with fine mechanical suspensions, and has been employed technically for clearing turbid liquids, and more recently for drying pasty mixtures, such as alizarine paste and ground peat; currents of high potential being used. It is, however, very marked in colloid sols, and closely simulates actual electrolysis. Spring (*Bull. Acad. Roy. Belg.*, 1898, [3], xxxv., 780) and Lottermoser ("Anorganische Kolloide," p. 76), and others give the following lists as to the behaviour of particles in aqueous sols:—

Wander to Cathode (+).	*Wander to Anode* (−).
Metallic hydro-oxides generally.	Colloidal metals.
Methyl violet.	Metallic sulphides.
Methylene blue.	Silver halides.
Magdala red.	Sulphur.
Titanic acid.	Selenium.
Silica in alkaline solution.	Aniline blue.
	Indigo.
	Eosin.
	Fuchsin.
	Mastic.

Many organic colloids, and some mineral ones (*e.g.*, silicic acid), wander to the anode in alkaline and the cathode in acid solution, while certain neutral substances in water, and many others in suitable mixtures of water and alcohol, are electrically indifferent. Definite charge in relation to the medium is not therefore an essential to the colloid state, though it appears to be so to electrolytic flocculation.

From what has just been said, it may be concluded that colloidal solutions must possess a sort of pseudo-electrolytic conductivity, but if this if the case, the amount is so slight that it is difficult to decide whether it depends on the colloid particles or on the residual traces of electrolyte, from which it is impossible to free the sols, as it but slightly exceeds that of the purest attainable water. Undoubtedly the particles do carry charges; but the charges are small, and the motion slow, and the number of moving particles very small compared to the ions in an ordinary electrolyte. It is also uncertain whether the particles are actually discharged at the electrodes, though occasionally particles appear to be repelled from both poles, and to collect in the middle of the liquid, which may be due to particles which have reached the electrode and received a contrary charge, wandering backwards and encountering and attracting originally charged particles proceeding in the opposite direction.

All inorganic sols are flocculated and precipitated by the addition of electrolytes, though they vary in sensitiveness. Organic sols are much less sensitive in this respect, but are frequently flocculated by the addition of organic solvents such as ether or alcohol, to which inorganic sols are usually indifferent. The precipitate is usually termed the "gel," but the term must not be confounded with "jelly" to which many of the gels are but distantly related. Gels may be "reversible," and may readily return to the sol state on restoring the original conditions, such as removing the added electrolytes; but frequently they are "irreversible," that is, the sols cannot be restored without going through the often complicated operations by which they were originally produced.

While the phenomenon of flocculation is apparently similar in inorganic and organic sols, it probably often differs rather widely in its causes, that of inorganic sols being closely connected with the electric charges of the particles; while in organic sols, osmotic effects which will be best discussed in connection with the properties of jellies, have probably a preponderating influence. Organic sols which have received definite charges usually behave with electrolytes like the inorganic.

Spring has pointed out that "coagulation" (flocculation and precipitation) takes place in two stages, though both may proceed simultaneously. In the first, the particles flocculate or adhere in larger aggregates; and in the second, these precipitate or settle under the influence of gravity. The rapidity of this second stage is much influenced by the size and density of the aggregates and the viscosity of the liquid, while the first stage is more clearly marked by the beginning of turbidity or opalescence, and, though not usually instantaneous, is better suited to observation.

Numerous experiments have shown that in order to produce flocculation, the concentration of the electrolyte must reach a certain minimum value ("Schwellenwert"), which varies with the electrolyte and the sol from a mere trace to a considerable amount.

Below this value, lapse of time produces no change, but, once it is passed, the sol is rapidly or slowly precipitated. In each case that ion of the electrolyte which has the opposite charge to the particles of the sol, has the determining effect, which increases rapidly with its valency. It has been stated that with divalent and trivalent ions it is proportional to the square and cube of that of monovalent, and Whetham (*Phil. Mag.*, [5], xlviii., 474) has shown that such a relation can be accounted for on the theory of probabilities with certain assumptions as to the electric charge required for coagulation; but the agreement of the observed numbers with each other, and with those required by theory, is not sufficiently close to make such speculation of much importance. It is also evident that the proportion will vary with the numerical value assumed for the monovalent ion. Perhaps more important is the observation of Hardy (*Proc. Roy. Soc.*, 1899, lxvi., 110) that the specific conductivities (ionic dissociation) of acid solutions exerting equivalent coagulating powers on negative and of alkaline on positive colloids are approximate constants; while acid solutions on positive and alkaline solutions on negative colloids have very varied values. This probably simply means that free H and free OH ions each have constant precipitating values for the colloids of opposite sign, while the acid and basic ions act according to their nature and valency.

It has been stated by Spring (*Rec. Trav. Chim. Pays-Bas.*, 1900, xix., 204) and others that the superior influence of polyvalent ions was not due directly to their valency, but to the usually greater hydrolytic dissociation of their salts, and, though this has been contradicted, it is not probable that the different ionic concentration produced by hydrolysis is without influence. Jordis considers that the colloid state and its phenomena are due to complex chemical compounds (*Zeit. f. Elektr.*, 1904, x., 509; *Sitzungsber d. Phys.-med. Soc. Erlangen*, 1904, xxxvi., 47; *Zeit. f. Elektrotech.*, 1904, x., 509; and Kanter, *Zeit., Anorg. Chim.*, 1903, xxxv., 16).

When a colloid sol is precipitated by an electrolyte the resultant gel always contains a small and apparently definite quantity of the precipitating ion of opposite sign to the particles, which cannot be removed by washing without restoring the gel to the sol condition or destroying its colloidal character. This quantity is proportional to the equivalent weight of the precipitating ion, and may, in many cases, be substituted by another ion of the same sign by washing with the appropriate salt (Sinder and Picton, *Journ. Chem. Soc.*, 1895, lxvii., 63). In each case an equivalent quantity of the non-precipitating ion is set free, and in the case of the stronger acids and bases may be estimated in the residual liquid by titration. These phenomena are not confined to inorganic colloids, but are very marked in many organic colloid precipitations, and apparently the same laws hold good in many pseudo-solutions, such as that of clay or of gum-mastic in water, which are generally distinguished as mere mechanical suspensions. The phenomenon of the retention of a

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

portion of the precipitating ion with the precipitate is well known in many ionic reactions, and is usually termed "co-precipitation" or "adsorption." It is, in fact, common to all bodies of largely extended surfaces, and to jellies, though the question whether in the last case the action is surface, or extends throughout the substance, must be discussed later. Even with solids, this is still, to a certain extent, an open question. Davis (*Trans. Chem. Soc.*, 1907, xci., 1666) has shown that in the absorption of iodine by carbon, an equilibrium is established with the surface action in a few hours, but that a further absorption goes on for weeks or months, which he considers due to a solid solution. The quantity of absorbed or adsorbed substance varies with the concentration of the solution with which it is in equilibrium, relatively more being taken from weak than from stronger solutions. The laws by which it is governed show close relations to those of solution-equilibria, and the phenomenon might perhaps be not improbably described as surface-solution. If a body be brought in contact with two immiscible solvents, it distributes itself between them in a ratio which, so long as its molecular condition is the same in both solvents, is, in accordance with Henry's law, a constant fraction dependent on its relative solution-pressure in the two solvents; or if C_a and C_b be its concentration in the solvents a and b and β a constant, $C_a = \beta C_b$. If, however, its molecular weight in the solvent b is n times as great as in a, the equation takes the form $C_a = \beta C_b^{\frac{1}{n}}$. Written in a general form, where x is the weight of the absorbed substance and m that of the absorbent, while C_b is the concentration of the solution, the equation becomes $\frac{x}{m} = C_a = \beta C_b^{\frac{1}{p}}$ (see Note), where β and p are constants to be determined experimentally, and represents very appoximately most adsorption-equilibria; but it is evident that p cannot represent the molecular weight in the ordinary sense since it is seldom a whole number, and usually indicates greater complexity in the solvent than in the adsorbent, which is impossible, when, as in the case of most electrolytes in very dilute solutions, the dissolved body is not merely monomolecular, but almost wholly ionised (*cf.* Walker and Appleyard, *Trans. Chem. Soc.*, 1896, lxix., 1334). The exact physical significance of p therefore remains to be determined, but where the case is one of surface-action $\frac{x}{m}$ is obviously an arbitrary expression, which is proportional for any one adsorbent in a uniform state of division, but gives no indication of the absolute surface involved. Even if the surface is molecular, as it probably is in jellies, we have little knowledge of the perplexity of the molecules. It can hardly be doubted that adsorption is dependent on the same forces as solution, as it is obviously selective and influenced by the chemical relations of the adsorbed and adsorbing substances, and in some cases is a preliminary to actual chemical combination.

(Note.—The exponential character of a curve of experimental results is easily tested by plotting the logs, either natural or common. If the logs of the equation $C_a = \beta C_b^{\frac{1}{p}}$ be taken, we obtain $\log C_a = \log \beta + \log C_b + \frac{1}{p}$. Hence, if we plot the experimental logarithmic concentrations of the solution as abscissæ and those of absorbed substance as ordinates, we shall, if the reaction is rigidly exponential, get a straight line, of which the slope (tangent) is $\frac{1}{p}$, and which cuts the vertical line, passing through the origin $\log C_b = 0$ (or $C_b = 1$) at $\log \beta$. It must not be forgotten in plotting logarithms that in fractional numbers the indices are $-$, while the mantissæ are always $+$. Exponential curves rise rapidly at first, but afterwards much more slowly and approximately to a straight line).

Freundlich ("Ueber die Absorption in Lösungen," *Habilitationsschrift*, Leipzig, 1906) has suggested a somewhat different empirical formula from the above for expressing adsorption, the so-called λ formula, in which λ for a given original concentration of the adsorbed solution is constant and independent of the quantity of adsorbent, in which case λ takes the place of β in the simple equation above expressing Henry's law. If, however, the concentration of the solution (*i.e.*, the relation of the dissolved substance a to the volume of the solution v) is varied, $\lambda = a\left(\frac{a}{v}\right)^{-\frac{1}{n}}$, where a and n are constants dependent only on temperature and the nature of the dissolved substance. This formula has been severely criticised by Bain (*Trans. Chem. Soc.*, 1907, xci., 1683), who shows that in some cases it leads to quite anomalous results. Freundlich adopts the view of Lagergreen (*Bihang t. Svenska Vet. Ak. Handl.*, 1899, xxiv., II., Nr. 4, 49; *Zeit. Phys. Chem.*, 1900, xxxii., 174), that adsorption is due to surface-tension, and points out that a dissolved body which lowers the surface-tension between solution and solid must become concentrated on that surface. That this is an actual factor in adsorption admits of no doubt; but our knowledge is not yet sufficient to enable us to calculate its magnitude; and it would seem to imply that adsorption was independent of any direct relation between the adsorbed and adsorbing body, which does not appear to be the case. It may be, however, that chemical relations between the solid and the dissolved substance influence its effect on the tensions of the solid-liquid surface, and so affect the results.

Inorganic sols are frequently coagulated by freezing or evaporation, and this is often really due to the traces of electrolytes always present in the sols, which become concentrated in the remaining liquid till they reach the minimum necessary for flocculation.

Colloid sols are not only flocculated by ions of opposite charge, but by other sols, the two colloids precipitating together in a way which closely stimulates an ionic reaction, and no doubt many of the precipitations, such as those of tannin and gelatin, tannin and basic colours, and basic with many acid colours are of this character. If the two sols are present in exactly the right proportions the precipitation is complete, and the precipitate of almost constant composition; but if one be in excess neither is completely precipitated. Probably in this case, as in flocculation by electrolytes, there is a minimum concentration below which precipitation does not take place.

The cause of this minimum concentration being needed is not clear, but it seems to indicate a certain initial resistance which must be broken down, and which is probably connected with the surface-tension between the particles and the liquid, or possibly with the stability of the electrical double layer, with which the surface-tension is closely connected as shown by the work of Quincke and Lippmann. The resistance to electrolytes is somewhat affected by the rate at which they are added, being greater when the addition is slow or in small quantities than when the necessary amount is added at once.

It can hardly be doubted that flocculation, whether by added electrolytes or by other colloids, is dependent on the neutralisation of the charges of the particles. In the case of electrolytes the ions, of which the charges are much greater than those of the particles, probably act as nuclei round which a number of particles are aggregated. The greater efficiency of polyvalent ions may thus depend on the larger aggregates which their larger charges are able to neutralise, and which naturally fall sooner under the influence of gravity and of cohesive attraction. As the particles probably owe their charges to attached ions, both flocculation and adsorption are in a sense chemical reactions. As one ion probably neutralises many particles the quantity carried down with the gel is relatively very small.

The action of "protective" colloids has been already mentioned, and is usually most marked in the effect of organic colloids on inorganic sols, but certain inorganic sols have also protective effect. Biltz (*Ber.*, 1902, xxxv.,

4431) found that zirconium hydroxide exerted a protective action on gold sols even stronger than that of gelatin; and Ruer (*Ber.*, 1905, xliii., 85), that zirconium and ferric sols containing traces of chlorine gave no precipitate with silver nitrate, the silver chloride remaining in colloidal solution. Kuspert has also produced gold and silver sols by reduction in presence of silica jelly. The efficiency of protective colloids extends to both positive and negative sols, though usually in different degrees, and is marked in a large number of organic colloids, many of which it may be remarked, are amphoteric, and take different charges according to the acidity or alkalinity of the medium. Thus gelatin and its peptones are among the most powerful with regard to gold sols, less than 1 mgrm. being sufficient to protect a litre of gold sol from precipitation by salt, while 80 mgrms. were required to exert an equal protective effect on a (much more concentrated) As_2S_3 sol. Most other colloids are less powerful in their effects, 10 to 20 mgrms. of egg albumen, a somewhat larger amount of gum arabic, and from 0·6 to 2 mgrms. of dextrine or starch being required to produce on gold sol an equal effect to 1 mgrm. of gelatin. Zsigmondy has taken advantage of this difference of effect for the detection and identification of colloids in mixtures (*Zeit. Anal. Chem.*, 1902, xl., 697). He calls the number of mgrms. of a colloid required to protect 10 cc. of a stable gold sol containing 0·0053 to 0·0058 per cent of gold from the precipitating action of 1 cc. of a 10 per cent salt solution the "gold value" of the colloid.

The cause of protective action is not fully explained. It is not due to the viscosity of the colloid, since the effect is produced by quantities too small to have any appreciable influence either on the viscosity of the liquid or the pedetic motion. Neither does it appear due to direct influence on the charges of the particles, since many colloids protect both positive and negative sols. It is more probably due to an actual coating of the particles of the sol with the protective substance. Bechhold ascribes this to surface-tension (*Zeit. Phys. Chem.*, 1904, xlviii., 385). Quincke has shown that in a system of immiscible liquids, A, B, and C, if the sum of the surface-tensions between A—B and B—C is less than that between A—C, B must spread between A and C, forming an intermediate layer (*Ann. Phys.*, 1888, [4], xxxv., 580). Bechhold shows numerically that this is the case in the system mastic-gelatin water, and that the mastic particles must therefore become coated with gelatin, which acts as a protective colloid to a mastic suspension. It is obvious that as the gelatin-coated particles have greater attraction for water and less for each other as compared to mastic they will have less tendency to coalesce. It may also be suggested that the gelatin may form a permanent double layer in the electric sense, which will render the neutralisation of the particles by oppositely charged ions difficult or impossible.

Platinum and gold sols exert a strong catalytic influence similar to that of platinum black and spongy platinum, decomposing hydrogen peroxide, causing combination of oxygen and hydrogen, and other similar effects. These seem merely due to the extended surface and very fine division of the metals, and belong rather to the general theory of catalysis than to that of the colloid state. Metallic sols have, however, been styled "inorganic ferments" from the analogy of their action to that of the catalyses, which are also colloidal, and which bring about similar reactions. Like them the action of the metallic sols is favoured by warmth, and in the case of hydrogen peroxide by small additions of alkali, while larger quantities slow or prevent it. In both cases certain substances, such as hydrogen sulphide and cyanide, exert a "poisonous" or inhibitory effect on the catalyst, which if slight is slowly recovered from. It is doubtful, however, if the resemblance is more than analogy.

The colour of metallic hydrosols is often intense, and varies with their condition, and especially on approaching flocculation; that of silver from dark brown to brown-violet, changing to deep green before flocculation. Gold hydrosols vary from intense red to blue-violet, taking the latter tint before precipitation by electrolytes, though some blue-gold sols are stable. The change of colour does not depend on the average size of the particles, but apparently on their aggregation. Kirchner and Zsigmondy (*Ann. Phys.*, 1904, [4], xv., 573) found that a red-gold sol rendered stable by gelatin becomes blue on evaporation to dryness, and under the microscope shows intensely coloured masses of many submicrons in a colourless medium. On moistening or re-dissolving these become again distributed, and the red colour is restored. Planck (*Ann. Phys.*, 1900, [4], i., 69) has shown that the light absorbed by small particles, which act as "optical resonators" to the electro-magnetic light-wave, must become redder, and that transmitted bluer as they approach each other. Seen in the ultra-microscope the gold particles usually reflect yellow light.

It has been noted that the precipitated gels of inorganic colloids are, in the opinion of the writer, not identical in structure with the true inorganic jellies, but at least in many cases they possess strong points of resemblance, and from their fine state of division retain many colloidal properties, most marked, of course, when they are reversible. They usually retain a portion of water with great obstinacy, so that it has often been mistaken for water of true chemical hydration. In some cases, however, the gels are probably true hydrates, which retain additional water by their surface-attraction. Van Bemmelen, Bütschli, and others have assumed various netted, cellular, and micellar structures in gelatinous gels, and to a certain extent these must have a real existence, though the extension of the theory to the true organic jellies is of doubtful validity. Von Weimarn (*Zeit. Chem. u. Ind. Kolloide*, 1907, ii., 76) has shown that gelatinous precipitates are obtained when concentrated solutions of crystalloids are mixed which react with each other to produce a compound which is very insoluble in the liquid; as, for instance, when concentrated solutions of barium thiocyanate and manganese sulphate are shaken together. These "jellies" may even be perfectly clear and transparent, and consist of drops of one or other solution, surrounded by a thin film of practically colloidal precipitate, which prevents coalescence and further reaction with the surrounding liquid. A very similar effect has been observed by the writer when a concentrated solution of calcium phosphate in hydrochloric acid is mixed with strong ammonia, when the mixture, though smelling strongly of ammonia, may yet contain acid calcium phosphate in excess, which is only slowly decomposed by the diffusion of ammonia into the isolated droplets.* The colloid particles are necessarily extremely insoluble in their media, and whenever precipitation occurs by the mixture of a precipitating solution the gel will be likely to take such cellular forms the dimensions of which will depend on the conditions of precipitation. In other cases where the precipitation is more gradual the particles will still adhere to loose amorphous masses, presenting a very large surface to the liquid, and it is probable that where dilute colloid solutions coagulate in which the surface-tension between the particles and water is considerable they will adhere to a more or less molecular network, which will contract in order to lessen surface and tear into flocculent masses, entangling and carrying down with them, as albumen does, all suspended particles in the liquid. Bütschli ("Untersuchungen über Mikroskop'sche Schäume und das Protoplasma," Leipzig, 1892; Verh. des naturh.-med. Vereins zu Heidelberg, N.F., 1892, v., 28—41, 42—43; 1894, v., 89—102, 230—292; 1896, v., 457—472) found that many gels showed network structures under the microscope with magnifications of about 2000 diams. when suitably treated, and detected similar structure in gelatin when hardened with alcohol or chromic acid; but

* Von Weimarn holds that the sole difference between the colloid and crystalloid condition lies in the extreme insolubility of the minute crystalline elements first formed, which prevents their uniting to form larger crystals, and considers that all matter is potentially crystalline.

as the unhardened gelatin appeared quite structureless it is very probable that the cavities were produced by the hardening process, and it may be noted that gelatin rapidly hardened by dehydrating agents is always more or less opaque and non-homogeneous.

The drying of gels is a continuous process, proceeding rapidly at first with vapour-pressures not materially below that of water, but gradually becoming slower, and the vapour-pressure lower as dryness is approached. Owing to the structure of the gel some time (days or weeks) is required for the mass to reach equilibrium with any definite vapour-pressure. The curve of vapour-pressures as compared with gradually diminishing percentage of water, though continuous, is not always regular, and does not appear to be exponential, and the process is usually irreversible with inorganic gels, or at least partially so, indicating a permanent change of state, though in some cases it may be connected with surface-tension, as in that of swollen gelatin drying in saturated water vapour, which will be mentioned later. Silica-jelly, which was very fully investigated by Van Bemmelen (*Zeit. Anorg. Chem.*, 1896, xiii., 233), may be taken as an example. The fresh gel was dried at a temperature of 15°, at which water has a vapour-pressure of 12·7 mm. in a series of exsiccators containing sulphuric acid diluted to known vapour-pressures until equilibrium was established. The fall of vapour-pressure was at first very gradual, but became more marked at 11 mm. when a water content of 3·7 was reached. (It is not clear from the diagram whether this is 3·7 mols. per mol. of SiO_2, or 3·7 grms. per grm., but probably the former). At a pressure of about 5 mm. and a water content of about 1·5 the gel, which had previously been transparent, became opalescent, and finally porcelain-white, and the curve ran horizontally; that is, the vapour-pressure remained constant until a water content of 1 was reached. This constant pressure indicates some change, chemical or physical, which takes place at this pressure, and if the proportions are molecular, might correspond to the decomposition of a definite hydrate, $(SiO_2)_2OH_2$, with a vapour-pressure of 5 mm., or to a change of form to one with lessened attraction for water. The opacity would also correspond to some definite change, as during the horizontality of the curve the gel would be a non-homogeneous mixture of the two substances or two forms. Below 5 mm. the gel again becomes homogeneous and transparent. Van Bemmelen supposes that at this point a further coagulation takes place with contraction of the gel and the production of air-spaces. Up to this point the curve is irreversible; that is, the gel will not absorb moisture in the same way, but only a much lessened quantity in saturated water vapour. Probably in the earlier part of the curve the surface-tension between the water and silica is different from its surface-attraction for water vapour, and the change indicated by the opaque state is not likely to be a reversible one. Below 4 or 5 mm. pressure the drying is a reversible one, and of the character common to all porous or finely divided substances. The drying of ferric hydroxide is very similar in character, but has no marked horizontal in its curve, which is throughout irreversible. From some, perhaps, rather fanciful resemblance of the curves produced to those caused by the hysteresis or "lag" in the magnetisation and demagnetisation of iron, the phenomenon has been described by that name. It produces some curious consequences, of which space does not permit discussion.

Most organic colloids seem to differ somewhat widely from the inorganic sols and gels which have been described, and in many cases their solutions, though possessing colloidal properties, approximate closely to what we should anticipate of true solutions of bodies of the high molecular weights which many of them are known to possess. They present more analogy to emulsions of immiscible liquids which become more miscible at higher temperatures than to suspensions of solid particles like the metallic sols, and have hence been called "emulsion colloids." Their sols usually show the Tyndall effect, and frequently, but not always, submicrons are visible to the ultramicroscope; but both may disappear with rise of temperature or the addition of a "common solvent." They are not usually sensitive to small additions of an elcctrolyte, and when "salted out" by larger quantities the effect seems often more osmotic than electrical. In some cases, however, when the colloid has acquired a definite positive or negative (ionic) charge it becomes much more sensitive to electrolytes, and where organic sols mutually precipitate each other the presence of a trace of electrolytes is probably essential to communicate the necessary charges. It is stated that gelatin free from inorganic constituents only gives a slight opalescence with tannin, and in ordinary cases the rapidity and completeness of the precipitation are much increased by the presence of salts.

Many aqueous organic colloid solutions are flocculated by additions of alcohol, ether, &c., and, conversely, alcoholic by addition of water, which have usually no action on inorganic sols. These effects are probably purely osmotic and dependent on the solubility of the substance in the different media, but such additions often change the electrical charge of the solvent.

In most cases organic sols are simply viscous liquids which on concentration become more and more viscous till they reach a practically solid form without any break of continuity; but others, usually by change of temperature, pass into the semi-solid state known as jelly. This solidification usually occurs with comparative suddenness, and is sometimes irreversible, but usually jellies have pretty definite melting-points at or slightly lower than their points of solidification, and varying only slightly with the concentration of the solution, but, like the melting-points of fats, not reaching their full value till some time after apparent solidification. Like crystallisation also, the change to the solid form is accompanied by evolution of heat, and liquefaction by its absorption.

Conversely, organic colloids in the solid form are capable of absorbing water or some other solvent, sometimes passing back without break to the viscous condition, but often reaching a maximum absorption and retaining the jelly state. Gelatin, for instance, dissolves direct to a viscous solution at temperatures above 25° or 30°, but swells to a jelly at lower ones, which melts if the temperature is raised. Cellulose and many of its derivatives and many animal and vegetable tissues swell, but do not dissolve without hydrolysis or other chemical change, and may be viewed as irreversible jellies. Thus, dyeing, tanning, and many other industries may be regarded as branches of colloidal chemistry.

The absorption of the solvent is accompanied by swelling or increase of volume, but especially in the early stages by marked contraction of the sum of the volumes of solvent and colloid and considerable evolution of heat; and, conversely, in dehydration heat is absorbed. Consequently, cold promotes swelling (as distinguished from solution), and heat has the opposite effect, though in dilute jellies, which can usually only be subjected to a small range of temperature, these effects are not very marked, and are often masked by other influences such as bacterial fermentation. As an illustration, it may be mentioned that gelatin is most rapidly and satisfactorily dissolved for culinary purposes by swelling in cold water, which may be thrown away with the dissolved impurities, and then melting the swollen jelly by setting the vessel in hot water.

(To be continued).

Notes on Two Methods of Preparing Monomethylamine. — Maurice François. — The author has investigated two methods of preparing monomethylamine, with special reference to the yield and purity of the product. Brochet and Cambier's method with formol and ammonium chloride does not give perfectly pure monomethylamine, and should not be used when purity is specially desired. On the other hand, Hofmann's method by brominated acetamide gives a very pure product, but the yield is comparatively low.—*C. R.*, cxlvii., No. 8.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 161).

The Iron Group (continued).

Procedure 65*c*.—To the H_2O_2 solution (*P. 65*b*), if coloured, add powdered Na_2SO_3 little by little until the solution is decolorised, and let the mixture stand twenty or thirty minutes. (White flocculent precipitate, presence of *titanium*).

Note.

1. This test serves to confirm the presence of titanium and to enable the quantity of it to be better estimated. H_2O_2 and the sexivalent titanium compound are rapidly reduced by the action of H_2SO_3, even in the cold, and titanium then precipitates as $Ti(OH)PO_4$. As in the case of zirconium, the precipitation takes place slowly. When there was only a faint colour with H_2O_2, no precipitate will be obtained, but 0·5 mgrm. of titanium is easily detected, if the concentration of the sulphuric acid does not exceed 1 cc. H_2SO_4 (1·20) in 25 cc. solution.

**Procedure* 65*d*.—Evaporate the first ether extract (*P. 65*a*) on a water-bath, and dissolve the residue in 3 to 5 cc. H_2SO_4 (1·20) and 3 to 5 cc. water. To the cold solution in a test-tube add 2 to 3 cc. 1 per cent KI solution and powdered Na_2SO_3 a little at a time until the iodine colour has permanently disappeared. (Yellow precipitate, presence of *thallium*). Filter, using preferably a hardened filter when the precipitate is small. Collect a little of the precipitate on a clean platinum wire, and introduce the wire into a colourless gas flame. (Momentary green flame, presence of *thallium*.

Notes.

1. The precipitation of thallium as TlI is practically complete, provided that the volume of the solution is small, say, less than 15 cc. The presence of iron does not prevent a good blank being obtained, nor does its presence in large quantity (500 mgrms.) prevent the detection of 0·5 mgrm. of thallium. TlI is readily oxidised to the soluble TlI_3 by iodine; therefore an excess of sulphite must be present.

2. Any quantity of thallium in excess of 10 to 15 mgrms. is precipitated in P. 11, except when the solution there treated with HCl contains the thallium in the thallic state.

3. The green flame test is a very delicate and characteristic test, but, on account of the volatility of the thallium compound, the green colour is seen only at the moment in which the wire is introduced into the flame. A hardened filter is recommended when the precipitate is small, because the precipitate can be more readily collected on the wire on account of the smooth surface of the filter.

Procedure 66.—Into the ammoniacal solution (P. 64 or P. 65) pass H_2S gas until the mixture after shaking blackens lead acetate paper held above it. (Black precipitate, presence of *nickel* or *cobalt*). Filter and wash the precipitate with water containing a very little $(NH_4)_2S$. (Precipitate, P. 67; filtrate, P. 81).

Notes.

1. In precipitating NiS, the use of H_2S has the advantage that the nickel is all thrown down at once, while with $(NH_4)_2S$ some of it usually remains in the solution, giving it a dark brown colour. If found more convenient, $(NH_4)_2S$ can, of course, be used, the filtrate being boiled to throw down the unprecipitated nickel, as described in P. 51.

2. The filtrate is, in general, tested for the alkaline earth elements, for these may be precipitated with the aluminium and iron groups when phosphate or certain other acid radicals are present, as discussed in P. 51, N. 6 and 7.

Procedure 67.—Transfer the H_2S precipitate (P. 66) with the filter to a casserole, and add 10 to 30 cc. of a cold mixture of 1 volume HCl (1·12) and 5 volumes of water. Digest in the cold for five minutes, stirring the mixture frequently, and filter.

Treat the residue by P. 68.

Boil the HCl solution until the H_2S is completely expelled, add 10 per cent NaOH solution until the mixture is slightly alkaline, transfer to a casserole, cool, and add 0·5 to 1 grm. Na_2O_2 a small portion at a time. Boil for several minutes to decompose the excess of Na_2O_2, and cool the mixture; filter off the precipitate, and treat it by P. 68, uniting with it the residue already obtained in the HCl treatment. Make the solution acid with acetic acid, warm it to about 60°, and pass in H_2S for two or three minutes. (White flocculent precipitate, presence of *zinc*), Apply to the precipitate the confirmatory test as described in P. 57.

Notes.

1. This treatment with dilute HCl serves to extract almost completely the zinc which may be present in this precipitate, owing to its having been carried down in the Na_2O_2 precipitate as described in P. 52, N. 11. A small proportion of the nickel and cobalt present (5 to 20 per cent) always dissolves in the dilute HCl, and the subsequent treatment with Na_2O_2 serves to separate them from the zinc. This separation is satisfactory when, as in this case, the nickel and cobalt are present in such small quantity that only an insignificant quantity of zinc is carried down with them. When therefore the H_2S precipitate is small, it may, instead of being treated with dilute HCl, be dissolved at once in *aqua regia*, and the solution treated directly as described in the last paragraph of the procedure.

2. This procedure must always be followed in order to determine whether or not zinc is present in the substance, except in the case that a satisfactory test for it has already been obtained in P. 57, or in the case that the original Na_2O_2 precipitate (P. 52) was small.

Procedure 68.—Transfer the residue insoluble in dilute HCl, and the Na_2O_2 precipitate (P. 67), with the filters to a casserole, add 5 to 15 cc. HCl (1·12), and a few drops HNO_3 (1·42), warm until the black precipitate is dissolved, and filter off the paper. Evaporate the solution nearly to dryness to expel most of the acid, add about 5 cc. water, and then NaOH solution drop by drop until the mixture is neutral, or until a permanent precipitate just forms. Test one-half of this mixture for cobalt by P. 69, and the remainder for nickel by P. 70.

Procedure 69.—To one-half of the neutral solution (P. 68) add 15 cc. 30 per cent acetic acid, and then 50 cc. 30 per cent KNO_2 solution; dilute to 100 cc., and allow the mixture to stand at least half-an-hour if no precipitate forms sooner. (Yellow, finely divided precipitate, presence of *cobalt*). Filter and wash with KNO_2 solution. If the precipitate is very small, incinerate the filter. Introduce a portion of the precipitate, or of the ash into a borax bead made in the loop of a platinum wire, and heat strongly, adding more of the precipitate or ash if no colour obtained. (Deep blue colour, presence of *cobalt*).

Notes.

1. The yellow precipitate is potassium cobalti-nitrite, $K_3Co(NO_2)_6$, which in solution dissociates into K^+ and the complex anion $Co(NO_2)_6^{\equiv}$. The precipitate is somewhat soluble in water, but very difficultly soluble in a concentrated KNO_2 solution, owing to the common ion effect of the potassium ion. In the formation of this substance the cobaltous salt is oxidised to the cobaltic state by the nitrous acid displaced from its salt

* From the *Journal of the American Chemical Society*, xxx., No. 4.

by the acetic acid, the cobaltic salt combining as fast as formed with the potassium nitrite, according to the equations :—

$Co+++(NO_2-)_2+2HNO_2=Co+++(NO_2-)_3+H_2O+NO$

and—

$Co+++(NO_2-)_3+3K+NO_2-=K_3Co(NO_2)_6$.

2. The formation of the $K_3Co(NO_2)_6$ precipitate takes place slowly, but even when very little cobalt is present (0·1 to 0·2 mgrm.) a distinct test is obtained within ten minutes, but the complete precipitation of a large amount of cobalt requires several hours, so that the method is ill-adapted for the removal of cobalt before testing for nickel. Moreover, when nickel is present, some of it is carried down with the cobalt, and this is true even when the total amount of nickel is small. For these reasons the test for nickel is made in a separate portion.

3. Nickelous salts are not oxidised by nitrous acid, and are not precipitated by KNO_2 except in a very concentrated solution, when a dark yellow to dark red precipitate of potassium nickelous nitrite, $K_4Ni(NO_2)_6$, may separate. By making the volume large, as directed in the procedure, there is do danger of the precipitation of nickel.

Procedure 70.—To the remainder of the neutral solution (P. 68) add 10 per cent KCN solution, a few drops at a time, until all or nearly all of any precipitate that may form at first re-dissolves; then add 0·5 to 3 cc. more (according to the amount of the KCN precipitate). Heat to 50° or 60° in an open casserole, with frequent stirring, for five minutes, or longer if the solution has not become light coloured. Filter off, and reject any small precipitate that may remain. To the filtrate, preferably in a test-tube, add freshly prepared concentrated NaBrO solution (see Note 3) until a piece of filter paper moistened with KI and starch solutions, when dipped into the solution, is coloured blue or brown. Allow the mixture to stand five to ten minutes, and filter. (Brown to black precipitate, presence of *nickel*).

Wash the precipitate; dissolve a small portion of it, if it is large, in 2 to 3 cc. HNO_3 (1·20); add 3 to 5 cc. 10 per cent tartaric acid solution, neutralise with 10 per cent NaOH solution, and add 3 or 4 cc. in excess. Pass in H_2S for about one minute, filter out any precipitate that may form, and saturate the filtrate with H_2S. Filter again if there is a precipitate. (Brown coloration, presence of *nickel*.)

Notes.

1. The reactions involved in the first test for nickel are as follows :—When a little KCN is added to the neutral solution, precipitates of (green) $Ni(CN)_2$ and (dark brown) $Co(CN)_2$ result except when only small amounts of these elements are present. The addition of more KCN causes the precipitate to dissolve owing to the formation of soluble complex cyanides as $K_2+[Ni(CN)_4]=$ and $K_4+[Co(CN)_6]==$. The complex nickel salt is stable in the air, but the cobalt salt is very readily oxidised according to the equation :—

$2K_4+[Co(CN)_6]==+O+H_2O=$

Potassium cobaltocyanide.

$=2K_3+[Co(CN)_6]\equiv+2K+OH-$.

Potassium cobalticyanide.

The first action of the NaBrO is to decompose the excess of KCN, chiefly with formation of KCNO. It then oxidises the nickel to the nickelic state, whereupon the nickel is immediately precipitated as brownish black $Ni(OH)_3$ by the NaOH present. The cobalt, though already in the cobaltic state, is not precipitated as $Co(OH)_3$, because the complex ion, $[Co(CN)_6]\equiv$, is so slightly ionised into its constituent-ions (Co+++ and 6CN−) that the concentration of the Co+++ does not suffice to produce with the OH− present the solubility-product for the $Co(OH)_3$, and that of the CN− is so small that it is only very slowly oxidised by the NaBrO.

2. In executing this procedure the following precautions should be observed:—A very large excess of the strong KCN solution over that required to re-dissolve the precipitate should not be added, for the excess must be destroyed by the NaBrO before the nickel can be oxidised and precipitated by it. Yet there must be sufficient KCN added, not only to combine with all the cobalt, but to furnish a moderate excess in order that the oxidation to the cobalticyanide may take place rapidly. Care must also be taken to heat the solution long enough in the air to complete this oxidation, before the NaBrO is added, for otherwise the latter reagent after destroying the free KCN will oxidise the decomposable cobaltocyanide with precipitation of $Co(OH)_3$, just as it does the nickelocyanide; the completion of the oxidation by the air is indicated by the disappearance of the dark colour in the solution. Finally, one must make sure (by applying the iodide-starch test) that an excess of NaBrO over that required to oxidise both the cyanide and the nickel has been added. If these precautions are observed, there is no difficulty in securing a precipitate with 0·2 mgrm. of nickel nor in causing 300 mgrms. of cobalt to remain entirely in solution.

3. The hypobromite reagent is prepared by adding liquid bromine to a known volume of 10 per cent NaOH solution until the solution becomes distinctly red, owing to the presence of excess of bromine, and then adding half as much more NaOH solution. This solution may be filtered through a hardened filter. It decomposes fairly rapidly, with formation of bromate and bromide, and also with evolution of oxygen, and should therefore not be used when more than a few days old. Instead of this reagent, bromine water and NaOH solution may, of course, be used. The concentrated hypobromite solution is recommended because a much smaller volume of it is required to decompose the large excess of cyanide used in the procedure.

4. When an alkaline tartrate solution containing a small amount of nickel (even 0·1 to 0·2 mgrm. in 20 cc.) is saturated with H_2S a clear brown solution is obtained, With somewhat larger amounts of nickel (10 to 20 mgrms.) the liquid is opaque but runs through a filter very readily. The condition of the nickel in this solution is not known. The presence of the tartrate serves merely to prevent the precipitation of $Ni(OH)_2$ by the NaOH solution, owing to the formation of a complex salt containing the nickel in the anion. The brown colour does not appear until the alkaline solution is nearly saturated with H_2S, so that care must be taken to use an excess of H_2S.

5. This confirmatory test for nickel is not interfered with by moderate amounts of other elements of this group, such as cobalt and iron (which, however, should not be present at this point), for on leading H_2S into an alkaline tartrate solution containing these elements, they are completely precipitated as sulphides, and may be filtered off, yielding a filtrate which in the absence of nickel remains clear when saturated with H_2S or becomes dark brown when it is present in even small amounts.

(To be continued).

CORRESPONDENCE.

SULPHATE OF AMMONIA.

To the Editor of the Chemical News.

SIR,—It is now some years since commercial sulphate of ammonia as used for manure contained any appreciable quantity of sulphocyanides. Indeed, the analyst had seldom any reason even to test for the presence of these compounds, for the sulphates of commerce have been so pure as to practically preclude the presence of cyanides. However, for some time past many samples which have come into my laboratory have not been satisfactory as regards colour, and I was induced to make a study of them to determine whether they contained sulphocyanides. In

some cases I found these to be present, but not in so large a quantity as would account for the coloration of the sample. Further examinations revealed the presence of organic compounds the nature of which need not be entered into here, but which in my opinion would be injurious to plant life. It must not be forgotten that sulphate of ammonia in itself is not a direct manure; before it can be utilised by plants it must undergo nitrification. The nitrifying organisms are probably, like most bacteria, susceptible to the influence of organic compounds —coal-tar derivatives—and would therefore be prevented from carrying on their work. These conclusions were not made known to the users in the West Indies of the manure, but they were subsequently confirmed by the managers of the estates, who reported that the manure had failed to give the usual results.

Still further evidence came to show that these compounds were injurious to plant life. It is customary to add a little sulphate of ammonia to the fermenting molasses to supply nitrogen to the yeasts, &c. Gas sulphate of ammonia was found to have very uncertain action and at times apparently an injurious effect on the fermentation. Bone sulphate was therefore substituted, with satisfactory results. It is, however, well known that bone sulphate has a very dark colour, so it was necessary to try and distinguish between the two colouring matters. My assistant, Dr. U. Bonelli, after some work has obtained a simple test. If a grm. of the sulphate is dissolved in 5 cc. water in a test-tube and strong ammonia added, an intense violet coloration is produced where the undesirable organic compounds are present, while no change occurs either with pure gas sulphate or bone sulphate. The detection of sulphocyanides in the presence of these organic compounds is not so satisfactory as in ordinary sulphate of ammonia. We have found the following method give satisfactory results. Five grms. of the sulphate of ammonia are moistened with strong ammonia and then dried in the water-oven. When quite dry the material is ground with 5 cc. of alcohol and the alcohol filtered off. This is diluted with water and tested with ferric chloride, and if sulphocyanides are present the usual bright red coloration is obtained.

I think it behoves all those who are employing sulphate of ammonia as a manure, either alone or as constituent of a compound, to make sure that what they purchase is both free from sulphocyanides and from the organic compounds which give the violet coloration with ammonia. One large firm in the city who are considerable users of this material have decided in future only to accept sulphate which passes these tests.—I am, &c.,

FREDK. J. LLOYD.

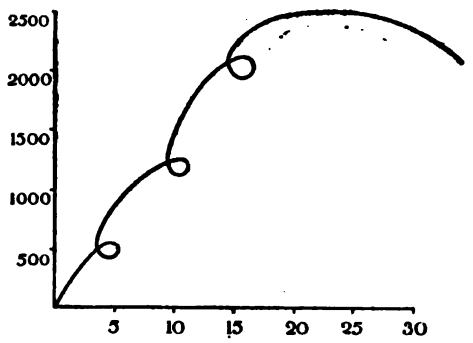
Laboratory,
6, Trinity Square, London, E.C.

WOMEN AND THE CHEMICAL SOCIETY.

To the Editor of the Chemical News.

SIR,—In attempting to take a share, however small, in this discussion I am labouring under two disadvantages, viz., (1) my great distance from the centre of disturbance; (2) the fact that I am not a member of the Society. The latter may be, perhaps, more of an advantage after all, as I am able to look quite impartially at the question. I should not have ventured into the field of strife, however, if it had not been for a letter which appeared in your issue of July 31st, signed H. Droop Richmond. I saw at once that Mr. Richmond had discovered the correct point of view from which to study the question, but, unfortunately, he did not fully work out the argument. I have taken the liberty and the trouble to do this to the best of my ability, and now present you with the result.

Mr. H. Droop Richmond's idea, like all truly great ideas, is very simple. Put shortly, it is this:—A woman member of the Chemical Society, by reason of being a woman, would have a better chance of getting her papers read and printed, and thus get a better opportunity for securing a lucrative post than a man. In other words, "the woman chemist would in the long run displace a man, another woman, and their children." Now, suppose 150 women were admitted as members each year. Of course it might be any other number, but 150 will do. These displace 150 men—married men with large families (I follow my authority in this, the "large" family being a sort of bonus); it follows then that these 150 unemployed chemists will not be able to pay their annual subs., and so will cease to be members. Thus each year more women will be admitted, and more *married* men displaced. What will happen? After so many years, say, five, large numbers of weddings will take place withing the ranks of the Society, now composed of only bachelors and spinsters. But these newly-married members will at once come under the law of displacement, so that eventually the Society will be composed entirely of *old maids* (with possibly a few confirmed bachelors). I believe this reasoning is correct; the statistics of the Registrar-General, and of the Chemical Society itself, make the average time of displacement of all married and marriageable members about twenty-five years. After this a reaction would set in, and in about another twenty-five years the Society would have returned to its present condition, and be ready to enter on another



"DROOPIAN" CURVE OF MEMBERSHIP OF THE CHEMICAL SOCIETY.

Ordinates = Women Members; Abscissæ = Years.

Notice flatness of curve for last seven or eight years, showing reluctance to part with with few remaining men members. The little rings on the curve show the periodic fluctuations caused by epidemic of weddings within the Society—wedding rings, in fact.

cycle. I have traced out a complete cycle of change on squared paper, and send it herewith. I cannot find a curve like it anywhere, so I have christened it the "Droopian." —I am, &c.,

E. G. BRYANT.

Port Elizabeth, Cape Colony,
August 21, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 5, August 3, 1908.

New Method of Making Pure Hydrogen.—M. Maurichéau-Beaupré.—When water is added to the product obtained by treating aluminium filings with powdered mercuric chloride and potassium cyanide, hydrogen is evolved, the reaction being accompanied by a gradual evolution of heat. The heat evolved can be used to regulate the rapidity of the decomposition. This method of preparing hydrogen (which may find application in aeronautics) has the following advantages:—It is very simple, the gas is pure, and the yield of hydrogen is very high for a given weight or volume of the metal.

No. 6, August 10, 1908.

Physico-chemical Analysis of Wines.—Paul Dutoit and Marcel Duboux.—The authors have shown that the method of determining electric conductivities, when applied to the analysis of the distillation products of wine, gives better results than acidimetry. Soda is added to the wine, which is then distilled. The curves of neutralisation by hydrochloric acid of the distillate are reproduced in the original paper.

No. 7, August 17, 1908.

This number contains no chemical matter.

No. 8, August 24, 1908.

Methylation in Ethylene Derivatives.—Louis Henry.—The replacement of H joined to C by the group $—CH_3$ in ethylenic glycols and hydrocarbons raises the molecular weight, diminishes the volatility, and raises the boiling-point. With the glycols which constitute the methylation series of ethylene glycol, $(HO)CH_2—CH_2(OH)$, the introduction of the $—CH_3$ group in place of hydrogen lowers the boiling-point. The boiling-points of the methylethylenic hydrocarbons increases regularly with successive substitutions of CH_3 for H. The hydrocarbons are monomolecular both in the liquid and gaseous states, while the hydroxyl compounds are monomolecular only in the gaseous state. The coefficient of association is lower the more completely the hydrogen is replaced by $—CH_3$, and the effect of increase of molecular weight is diminished by the simplification of the molecule $(CH_3—OH)_n$. The hydrochloric ethers are similar to the hydrocarbons, and the replacement of —OH by Cl in the alcohols is accompanied by a considerable lowering of the boiling-point. The oxides also appear to be monomolecular or very feebly associated compounds.

NOTES AND QUERIES.

Potential Energy of the Elements.—I would be sincerely obliged if any reader would afford me the following data for at least three elements, not including H, N, and F:—The quantity of heat in large cal. per grm.-mol. taken up in the change of an element from—(1) A solid state to boiling-point; (2) a solid state to its critical state. Also the density at B.P. and critical state referred to water.—Daniel J. Rankin.

Official Methods of American Agricultural Chemists.—Where can I obtain these Official Methods? How are the Bulletins issued, and what is their price?—X.

Rotting Cotton Thread.—I wish to place cotton thread into a solution and dry it so that it may be worked up in a loom along with ordinary cotton into fabric. After it is made I wish to pass the whole through heat or other dry process, so that it will rot away the cotton that has been through the chemical process and leave the ordinary cotton in its proper state. If any reader would kindly give me the names of the chemicals and particulars of working, or tell me where I can obtain the information, I should esteem it a great favour.—J. C. Rollinson.

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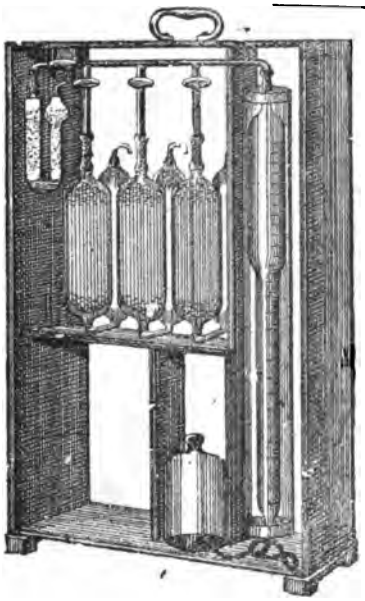

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THE CHEMICAL NEWS.

Vol. XCVIII., No. 2550.

THE FACTORS WHICH INFLUENCE THE RATE OF ALCOHOLIC FERMENTATION.*

By ARTHUR SLATOR, Ph.D., D.Sc.

The transformation of glucose into alcohol and carbon dioxide by the action of yeast is probably not a single chemical reaction but a series of reactions. If one reaction of the series proceeds relatively much more slowly than the others, then the velocity of the transformation is determined by the rate of this slow reaction.

Evidence is brought forward to show that the initial rate of fermentation by living yeast is controlled almost completely by one single reaction. By considering only these initial velocities certain disturbing secondary reactions are eliminated, for change of conditions, which are usually considered to influence the rate of fermentation, have no effect on these initial velocities. The simple laws governing the rate are evidence in favour of considering one reaction to have the control of the velocity.

The rate of fermentation is exactly proportional to the amount of yeast present. The rate of fermentation of the four fermentable hexoses (glucose, fructose, galactose, and mannose) is almost independent of the concentration of the sugar. Glucose and fructose are fermented at approximately equal rates. Yeasts which ferment glucose do not always possess the property of fermenting galactose, and this sugar is fermented only by yeasts which have been grown in the presence of galactose. The fermentation of mannose is similar to that of glucose, but the rates of the two reactions are not equal. The enzyme which ferments mannose seems to be more sensitive to heat than the one which ferments glucose. The influence of temperature on these reactions is almost the same in the case of glucose, fructose, and mannose, but rather less in the case of galactose. These results are most easily brought into accord on the assumption that the enzyme combines with the sugar. Glucose and fructose form the same compound; galactose and mannose form different compounds. It is possible that there exist three different enzymes: glucozymase, which ferments glucose and fructose; galactozymase, which ferments galactose; and mannozymase, which ferments mannose. It is probable that no compound has yet been isolated which can be considered intermediate in alcoholic fermentation. The suggestion that lactic acid is such a compound is of special interest, and has attracted much attention. Lactic acid does not ferment, or ferments only very slowly and incompletely. This is the chief objection to the hypothesis. An intermediate compound must be at least as reactive as the original reagent. The suggestion that lactic acid is liberated in a "nascent state" is considered unlikely, for in constructing the mechanism of other similar chemical reactions such suppositions have not been found necessary.

Fermentation by yeast-juice differs in many respects from that by living yeast. It is probable that the mechanism of the reaction is the same in each case; but the relative rates of the different steps in the two processes are different. The experiments show that there is an essential step in fermentation in which phosphates in some form or another play a part. This reaction proceeds too rapidly in fermentation by living yeast to have any controlling influence on the velocity; but in the fermentation by yeast-juice it proceeds relatively more slowly and influences the rate.

The method of investigating complicated reactions by considering only initial velocities has the advantage that disturbing side reactions are to a large extent eliminated. The method can probably be applied with advantage to other enzyme processes.

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

COLLOIDAL CHEMISTRY.*

By H. R. PROCTER, M.Sc.

(Concluded from p. 170).

The absorption of water vapour by most solid organic colloids is a cyclical and reversible process, the colloid returning by evaporation to its original condition, and it therefore follows that Clausius's equations can be applied in this case as well as in the expansion of bodies by heat. Rodewald (*Zeit. f. Phys. Chem.*, 1897, xxiv., 193) has done this in the case of starch, and has calculated from the results its internal pressure, which was found to vary from 2073 kilos. per sq. cm. in dry starch to 561 kilos. in starch fully saturated with aqueous vapour. He also calculated from its vapour-pressure its molecular weight in the solid form as 4370, closely corresponding to twenty-seven times the empirical formula $C_6H_{10}O_5$. This is interesting as derived in a different way from the molecular weight determinations in liquids. The method is obviously applicable to other solid colloids.

The pressure exerted in the early stages of colloid swelling are very large, though they become almost inappreciable as the maximum is approached. The ancient use of dry wood wedges subsequently moistened is an instance of this, and it may be mentioned that stones of the trilithon at Baalbec in Syria, weighing over 1000 tons, have obviously been split in this way. The effect has usually been ascribed to capillary contraction, but a little consideration will show that capillarity cannot produce expansive effects. A pile of thin plates (*e.g.*, cover-glasses on the microscope stage) will be compressed and not expanded if water is allowed to be drawn between them by capillarity. The effect is necessarily molecular and osmotic, the liquid getting *within* the sphere of molecular attractions, and so sustaining and liberating for expansion a portion of the enormous internal pressures which have just been mentioned (in the case of starch some 1500 atmospheres).

The point of maximum swelling, whether in liquid or vapour, is obviously a definite equilibrium which is reached when the attraction of the colloid for the liquid or vapour is balanced by the internal attractions of the colloid and the liquid in themselves. The liquid and the colloid are in complete osmotic equilibrium, and consequently, according to a well-known law, are both in equilibrium with the vapour. P. von Schroeder draws attention to an apparent exception to this law, which is of considerable importance, and for which he was unable to offer a satisfactory explanation (*Zeit. f. Phys. Chem.*, 1903, xlv., 75). He found that agar and gelatin jellies swelled to a considerably larger extent when immersed in water than they did in saturated aqueous vapour, and that when the jelly, saturated by immersion, was suspended in saturated vapour, it dried till it was again in equilibrium with the vapour; thus evidencing a greater vapour pressure than the liquid water with which it was in osmotic equilibrium. He found, however, that when the jelly was swollen with an N/100000 solution of a sulphate instead of water, no contraction, but slight further swelling, occurred; while an N/1000000 solution diminished but did not prevent the evaporation. As the N/10^5 solution may be assumed to be completely ionised, it will contain three ions in 2×10^5 cc., and at 0° C. will exert an osmotic pressure of 340 dynes per cm.^{2}. This would involve work of 340 ergs per grm. of water removed, and would correspond to the pressure of a column of water only 0·344 cm. in height, but Von Schroeder took special precautions to avoid difference in level. If, however, a

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

portion of water is removed from the mass the surface is extended, and work is done (on the assumption of spherical form) of 370 ergs. The surface tension would be less and the osmotic pressure greater, at laboratory temperature (about 365 dynes at 20°), so that the energy of surface tension agrees well with that found to produce the effect, and is a sufficient cause. The figures are perhaps less important as explaining an apparent anomaly than as illustrating the smallness of the forces involved in considerable changes of volume near the maximum swelling. The relation of surface tension to the drying of gels consisting of aggregates of particles, as that of silica, may be here again referred to. If the common tension between the surfaces of the particles is less than that of water itself, as in gels is necessarily the case, they will retain a large amount of water by surface attraction, the vapour pressure of which will be equal to or somewhat less than that of liquid water according to the tenuity of the film. As the water is removed by evaporation the particles will cohere by their mutual attraction, and if this cohesive attraction is greater than the difference between the tension of water and that of the common surface, or, in other words, than the difference between the attraction of the particles for water and the attraction of water for itself, water will not again spread between the particles, and the drying will be irreversible as in the earlier stages of the silica jelly.

The rate of absorption of liquid by solid colloids has been studied by Hofmeister (*Archiv. f. Exp. Path. und Pharm.*, 1890, xxvii., 395), and more recently by Pauli (Pascheles) (*Ibid.*, 1895, xxxvi., 100). The latter showed, both mathematically and by experiment on agar and gelatin, that if Q be the degree of swelling and M its mass, the rate of swelling $\frac{dQ}{dt}$ at any given time is equal to (M - Q)C, the difference between the actual and the maximum swelling multiplied by a constant, which varies with the material, and probably with the temperature. This is a law common to many chemical and physical changes, but complicated in this case by the constantly increasing thickness of the swelling colloid. For very thin plates, where this can be neglected, and between the times t and t_1 and the swelling Q and Q_1 the constant $C = \frac{1}{t_1 - t} \text{ en } \frac{M - Q}{M - Q_1}$, and for agar is about $9{\cdot}5 \times 10^{-2}$.

The swelling of gelatin jelly is much influenced by acids and alkalis, both of which largely increase both its amount and its rapidity. The action of neutral salts on swelling, as regards gelatin, has been investigated by Hofmeister (*Archiv. fur Exp. Path. and Pharm.*, 1891, xxviii., 210; their effect on melting and gelatinising temperatures by Pauli (Pfluger, *Archiv.*, 1898, lxxi., 1), and on viscosity by von Schroeder. All these effects are practically parallel; salts which lesson swelling raise the melting-point of jellies and increase the viscosity of the sols. The effect seems an additive one in which anions and cations take part, but no definite laws have been established. The order of the salts investigated, placing those first which most raise the melting-point, is sulphates, citrates, tartrates, acetates, water, chlorides, chlorates, nitrates, bromides, iodides as anions, and K, Ca, Na, Sr, and Mg as cations, and those placed before water raising and those after it lowering the melting-point. Whether the same order applies to other colloids than gelatin is uncertain, but no doubt the effects depend to a considerable extent on the specific affinities of salt and colloid.

The swelling, though mainly an osmotic effect, is influenced by the solid cohesion of the jelly, and there can be little doubt that at the moment of setting a network is formed, whether molecular or coarser, which remains intact during the existence of the jelly. Experiments made by the writer show that the volume of the jelly at the moment of setting has considerable effect on the maximum of swelling, though concentrated jellies after drying swell in water to more, and dilute to less, than their original volume. A solution originally containing 23 grms. of water reabsorbed 14·5 grms., one of 11 grms. 7·8 grms., and one of 5 grms. 6 grms. per grm. of dry gelatin. If weak jellies are mechanically squeezed a considerable amount of liquid, mostly water, can be expressed, and this has been used as an argument for the cellular (two-phased) structure of jellies. It is obvious, however, that near the swelling maximum this must be possible in any case, since it has been already shown that considerable changes of volume can be produced by changes of osmotic pressure equivalent to less than 1 cm. of water.

The phenomenon of "semipermeability" is frequently exhibited by colloid gels and jellies, and, in fact, all the usual semipermeable membranes are colloidal. Just as solids will dissolve in one solvent, and not in another, so jellies are selective in the liquids which they will absorb. Indiarubber will absorb hydrocarbons but not water; gelatin and agar, water but not hydrocarbons or alcohol; and the process of transfusion in a semipermeable membrane is not one of mere mechanical filtration of a finer order, but of absorption and solution on one side of the membrane, and solution and diffusion on the other, and is intimately connected with the chemical character of the membrane and with its ionic charge. Colloid jellies or membranes are usually impermeable to other colloids, while most dissolved salts diffuse in them with nearly the same rapidity as in pure water, though they are sometimes semipervious to those containing a common ion. In dilute jellies, however, some diffusion of finely divided colloids takes place; and Bechold (x. Vortr. 78, *Vers. d. Naturf. u. Aerzte*, Stuttgart, 1906) has used dilute jellies (under pressure, supported on coarser media) for the fractional filtration of colloid solutions. Gelatin and gelatinous membranes are semipermeable to alcohol, which exerts considerable osmotic pressure in a gelatin-lined cell, and jelly is easily dehydrated in absolute alcohol till it contains less than its own weight of water, very little alcohol diffusing into the jelly. On the other hand, if alcohol is mixed with a warm gelatin solution in insufficient quantity to cause precipitation and is allowed to set, the jelly will show much more than its normal maximum swelling when immersed in water, from the osmotic pressure of the contained alcohol which cannot escape. This observation proves clearly that the contraction is not due to any chemical action of the alcohol, but to simple osmotic causes. If alcohol in sufficient quantity be added to a warm and ungelatinised solution of gelatin, it causes separation of the latter, first as a milky liquid, which rapidly flocculates, and, if stirred, coheres into a solid mass, thus showing all the usual phenomena of coagulation, from what are obviously purely osmotic causes. Similar effects occur with gums, dextrine, and most other water-soluble colloids. With egg-albumen the coagulation is irreversible, but in most cases resolution takes place on again substituting water.

Dilute acids and alkalis greatly raise the maximum swelling of gelatin and all gelatinous animal tissues such as skin. French gelatin with a maximum absorption of seven to eight times its weight of water will absorb over fifty times in hydrochloric acid of 0·003 grm.-mol. per litre, at about which concentration the swelling reaches a maximum, falling gradually to below twenty times at 0·4 grm.-mol. per litre, much beyond which the experiment cannot be carried because of solution of the jelly in the acid. Similar effects occur with all other acids which the writer has investigated in which a sufficient concentration can be employed without causing solution. The concentration of the acid in the swollen jelly is always greater than in the external solution, increasing (in the case of gelatin at least) somewhat rapidly at first, before the maximum of swelling is reached, and later remaining nearly constant at 0·002 or 0·003 mol. per litre above the outside solution; while the total quantity of absorbed acid per grm. of gelatin increases very approximately by an exponential law. The whole of the hydrochloric acid can be estimated by titration with caustic alkali in presence of phenolphthalein, but only a portion with methyl orange, showing that one part of the acid exists in a less ionised

state than the remainder. The swollen jelly can no longer be dehydrated by absolute alcohol, showing that the osmotic pressure of water into the jelly is now greater than that into the alcohol, but a part of the acid, approximately corresponding to that estimated by methyl orange, can be removed by repeated treatment with alcohol. The jelly is still pervious to acid and salt solutions, though probably to a less extent than neutral jelly, and it is obvious that one portion of the acid exists in some sort of combination with the jelly, while another is merely osmotically absorbed. It is clear that no definite conclusions can be drawn until in some way these portions can be distinguished; and as a first approximation (obviously not quite correct) it was assumed that the acid absolved in swelling was of the same concentration as that of the outer solution, the less ionised and more constant portion, which may be called "fixed acid," being ascertained by subtracting the calculated value of the acid in the absorbed liquid from the total found in the jelly. The result showed that the "fixed acid" rapidly reached a maximum coincident with the concentration of maximum swellings, after which it remained approximately constant, a slight downward curve being probably due to the inaccuracy of the assumption that the strength of the absorbed solution was equal to that outside, or, what amounts to the same thing, that the whole of the water in the jelly was present as absorbed outer solution. With a small correction in this sense the amount of "fixed acid" obtained in this way closely corresponds to that not estimated by phenolphthalein, and incapable of being washed out by alcohol. From inspection of the curves it is obvious that that of total acid per grm. of gelatin is of an exponential type, but it is compounded of that of "fixed acid" the character of which strongly suggests chemical combination somewhat complicated by dissociation or hydrolysis, and the curve of swelling, which after its maximum is reached is of a hyperbolic character. If, as is probable from the empirical formula, the molecular weight of gelatin is about 1·800, or its multiple, it would roughly correspond to the fixation of two mols. of HCl by one of gelatin.

The action of salts on unacidified gelatin has been mentioned, and common salt causes a small increase of swelling as compared to water, and even saturated solutions have no dehydrating effect. If, however, salt is added to jelly swollen with hydrochloric acid, swelling is immediately reduced, and very powerful dehydration comparable to that of absolute alcohol is obtained by saturated salt solutions in presence of the amount of acid necessary to the maximum swelling effect. At the same time the total acid in the jelly is diminished, but the "fixed acid" is gradually, though not largely, increased. It is probable that if the concentration of the hydrochloric acid could be increased to an equal extent to that of the salt without causing solution, an equal dehydration would be produced, and it is difficult to resist the conclusion that the swelling is due to the chlorine ion in the acid gelatin, and is osmotically repressed by the increase of the same ion in the solution. Other acids with their neutral salts produce similar effects. The fact that similar dehydration is produced by other acids, *e.g.*, sulphuric, in conjunction with sodium chloride, and probably with other salts, does not contradict the conclusion that it is the common anion which is operative, since in the case suggested an equilibrium of sodium and gelatin sulphate and sodium and gelatin chloride would result. The cause of the general swelling effect of acids must, however, in some way be intimately connected with their common H ion, and that of alkalis, which is equally powerful, with the hydroxyl ion. The alkaline swelling is not repressed by sodium chloride, even when caused by sodium hydrate, but is repressed by sufficient concentration of the hydroxyl ion in the outer solution. The above are the summarised results of a research on which the writer has been some time engaged, and which he hopes shortly to publish with a more detailed discussion of the operating causes. It may be remarked that the dehydration of acidified gelatinous tissues has an important technical application in the preservation of sheep pelts by "pickling" with salt and sulphuric acid, and its bearing on numeral tannages in which salt is employed with alums and other compounds of acid reaction is very obvious. As gelatin is decidedly amphoteric, and is known to be capable of decomposing many salts in solution, it is quite possible that some of the complicated effects of unacidified salt solutions which have been referred to may be due to partial absorption of the acid of the salt and the osmotic effect of the remainder on the compound formed.

Space will not allow of any detailed discussion of the coagulation of the various albumins by heat and precipitating agents, which, important as they are from the physiological aspect, have not yet yielded many facts which can be explained on general principles. Albumins, carefully freed from electrolytes by dialysis, are electrically neutral and incompletely coagulated by heat or by those precipitants which do not at the same time communicate an electrical charge. The addition of a trace of acid which gives a positive charge renders them liable to complete coagulation by heat and much more sensitive to the action of electrolytes; but larger quantities of acid, perhaps by producing soluble acid-albumins, prevent coagulation. Traces of alkalis which give a negative charge have a similar effect, though the precipitation is usually less complete, and excess again prevents or lessens precipitation. Coagulation by heat is usually irreversible, and so also that by alcohol, though zymases are precipitated by the latter in a reversible (soluble) form. Salts of the heavy metals, even in small quantities, produce irreversible coagulation, very possibly connected with combination or colloidal precipitation with a colloidal compound of the metal. Excess of the precipitant frequently prevents precipitation, probably by the formation of new complex compounds. Possibly there may be some connection between these phenomena and the irreversible (tanning) effects of some metallic solutions on gelatinous tissues. These solutions are usually of "basic salts" containing metallic oxides colloidally dissolved. Precipitations of albumins by alkali salts, especially in neutral solution, are generally reversible. (The work of Cohnheim, "Chemie der Eiweisskörper," 1904, and of Pauli and Spiro in the *Beitr. z. Chem. Phys. und Path.* from 1903 onwards seems specially worthy of attention).

The relations of colloid chemistry to technology can here only be briefly alluded to. The industries of india-rubber, gutta-percha, gums, dextrine, glue and gelatin, and cellulose derivatives are of course all colloidal, and it is probable that many reactions which have been described as chemical may really be those of absorption compounds. Tanning and dyeing are particularly concerned with the mutual precipitation of colloids and that by inorganic reagents, and in these industries also the distinction between physical and chemical reactions in any given case is one of extreme difficulty, which is much accentuated by the want of any method for the separation of colloids comparable to crystallisation, or even fractional distillation.

Among industries with inorganic colloids may be mentioned the comparatively new one of the manufacture of colloid fibres for electric glow-lamps from difficultly fusible metals, and both earthenware and agriculture are largely dependent on the properties of colloidal suspensions. Glass manufacture is also a colloid industry, and modern pigments are largely colloidal precipitates and colour-lakes.

In conclusion it is only right to acknowledge indebtedness to the excellent work of Dr. Arthur Müller, "Allgemeine Chemie der Kolloide" (J. A. Barth, Leipzig, 1907), with its extensive bibliography; and to the *Zeitschrift für Chemie und Industrie der Kolloide*, edited by Dr. Wolfgang Ostwald, without which the preparation of such a report would have been extremely laborious. Owing to the very numerous and often somewhat inaccessible journals in which important work has appeared, it has been necessary in many cases to trust to abstracts, and it is possible that from this cause full justice has not been done to some authors.

THE INDESTRUCTIBILITY OF MATTER AND THE ABSENCE OF EXACT RELATIONS AMONG THE ATOMIC WEIGHTS.

By DANIEL F. COMSTOCK.

THE two chief reasons briefly stated for believing in the evolution of the elements one from another are, first, that some such process is undoubtedly taking place in the case of the radio-active substances, while we are being forced toward the conclusion that all the elements are radio-active to some degree; and, second, that in the hottest stars only two known elements occur, namely, hydrogen and helium, while as we pass successively to cooler and cooler stars the other elements gradually make their appearance in a more or less orderly manner. Apparently this can only mean that at these transcendental temperatures the forces due to molecular or atomic impact are comparable with the interatomic forces involved in the breaking up of one element to form another, and hence the combination necessary for the formation of the heavier elements can take place only after the temperature has sufficiently dropped.

There is one seemingly fatal objection, however, to any very simple statement of the evolutionary theory, and this objection has not been sufficiently emphasised. The difficulty is this, that so far as we know there are no exact simple relations between the various atomic weights, whereas if we are to assume, as the simplest form of the evolutionary theory does, that the lighter elements come from an atomic disintegration of the heavier ones, or *vice versâ*, it is evident that simple additive relations must exist.

As we know, many simple additive relations do exist, but they are approximate, not exact, and the deviations from exactness, though small, are larger than we can explain from error in atomic weight determinations.

On the basis of common conceptions, therefore, the evidence seems contradictory, certain facts seeming to require the simple evolutionary idea, while another fact, the inexactness referred to above, seeming to deny it.

I wish to show that on the basis of the electrical constitution of matter this inexactness is not only to be explained but it is to be expected.

By the electrical constitution of matter is meant merely that conception, which has grown in favour among physicists of late, which considers the atom to consist wholly or in part of a group of electric charges. Some of these make themselves evident in an ionised electrolyte, or in a gas, but in general the fact that the two electricities, positive and negative, are present in equal amounts makes the atom neutral as regards action on an outside point.

Now, since moving charges always act like currents, and hence must set up magnetic fields, it must follow that when an apparently uncharged atom is set in motion there will be set up inside of it a magnetic field, which depends for its strength and distribution on the number, position, and magnitude of whatever charges the atom may be supposed to contain. It will therefore require more energy to set the atom in motion because of the necessity of building up this field, and hence the atom will have an added inertia, *i.e.*, an added mass because of these charges.

Now, it has been shown mathematically by the author (*Phil. Mag.*, Jan., 1908), without making any assumption as regards the structure of the atom, that *this added mass due to the electric charges is strictly proportional to the total electrical energy contained in the atom.*

By electrical energy is meant the energy which we know must exist in space wherever there are electric lines of force. Two charges, one positive and one negative, attract each other, and if these are separated to a distance, work must be done against their mutual attraction. Since the charges themselves have changed in no way, the energy put in must exist in the surrounding space in a form generally known as "strain in the ether." Magnetic lines of force also correspond to energy stored in space, and the statement proved in the paper mentioned above is that the added mass which an atom possesses because of the electric charges which it is supposed to contain depends only on the total electric and magnetic energy which the atom contains, and is directly proportional to this energy.

The expression found may be written—

$$M = \frac{4}{3}\frac{E}{V^2},$$

where M is this "electric mass," V is the velocity of light, and E is the total contained electromagnetic energy of the kind described above.

The amount of energy which corresponds to a grm. mass is enormous. It is readily calculated from the formula, and is found to be 7×10^{20} ergs, approximately, so we may make the statement that, on the present bases, *the inertia of a grm. mass is due to the existence within it of* 7×10^{20} *ergs of confined energy.*

Now, when the atom of an element breaks up the process is a violent one and a large quantity of energy is lost, *i.e.*, goes ultimately into heat. Hence, if this electric energy lost—and on the basis of the electrical conception it *is* electrical energy—it follows from the above that *there must have been a loss in mass accompanying the atomic disintegration* because of the energy lost. Hence, taking the simple case of an atom of A, splitting up violently, and forming atoms of the elements B and C, we would find that the mass of B and C, taken together, would be a little less than the mass of A, from which they came, the difference corresponding to the loss of mass accompanying the loss in energy.

Now, the loss of energy when an atom of radium breaks up is known, and calculation shows that this would give a loss in mass which would be of about the same size as the deviations in atomic mass or weight so common in the table.

In an important paper, Rydberg (*Zeit. Anorg. Chem.*, 1897, xiv., 66) has shown that the atomic weights of the first twenty-seven elements of the periodic system approximate to whole numbers very much more closely than chance could bring about. He has also shown that the atomic weights of these elements are best considered as the sum of two parts (N + D), where N is an integer and D is a fraction, in general positive and smaller than unity. If M is the number of the element in the system (called by Rydberg the "Ordnungszahl"), then N is equal to 2M for the elements of even valence and 2M + 1 for the elements of odd valence. The accompanying table shows the various quantities. I have used, however, the International Atomic Weight values for 1907 instead of those Rydberg used.

Many besides Rydberg have, of course, noticed and studied the curious deviations in the table of atomic weights, and Rydberg's work is mentioned merely because it seems unusually explicit.

The orderly arrangement of the series is striking. It will be noticed that in three cases only are the D's greater than unity, and only in two cases are they negative.

Rydberg points out that although the heavier elements do not conform well to this scheme, *i.e.*, do not in general give the small fractional values of (D) noticed above, yet this is in reality no valid objection, for the numerical values of the weights of heavier elements depend much more on the value of the arbitrary unit chosen than do those of the lighter weight elements, and hence they can have little influence one way or the other in estimating the validity of the curious relations he sets forth.

The whole question is, of course, whether these differences represent real physical deviations from something, or whether they are merely mathematical remainders. Rydberg certainly believes them to represent physical realities, and considering the before-mentioned overwhelming improbability that the approximation of the atomic weights to whole numbers is due to chance, we can hardly doubt that he is right.

Sign.	M.	N. 2M.	N. 2M+1.	Atomic weight.	D.
He	2	4		4	
Li	3		7	7·03	0·03
Be	4	8		9·1	1·1
B	5		11	11·0	0
C	6	12		12·00	0
N	7		15	14·01	−0·99
O	8	16		16·00	0
Fl	9		19	19·0	0
Ne	10	20		20·0	0
Na	11		23	23·05	0·05
Mg	12	24		24·36	0·36
Al	13		27	27·1	0·1
Si	14	28		28·4	0·4
P	15		31	31·0	0
S	16	32		32·06	0·06
Cl	17		35	35·45	0·45
A	18	36		39·9	3·9
K	19		39	39·15	0·15
Ca	20	40		40·1	0·1
Se	21		43	44·1	1·1
——	22	44		—	—
——	23		47	—	—
Ti	24	48		48·1	0·1
V	25		51	51·2	0·2
Cr	26	52		52·1	0·1
Mn	27		55	55·0	0·0
Fe	28	56		55·9	−0·1

The question will doubtless be asked why there is no loss of mass found when a violent chemical reaction takes place and energy is lost. The expression for loss of mass in terms of loss of energy, when written in the differential form (substituting $V = 3 \cdot 10^{10}$), is—

$$\Delta M = \frac{4}{27} \, 10^{-20} \, \Delta E,$$

and if we substitute the heat of reaction of any known chemical reaction for ΔE, we find that ΔM is too small to be detected even by the delicate experiments of Landolt.

For the reaction $O_2 + 2H_2 = 2H_2O$, ΔM for 1 grm. molecule is about 10^{-10} of a grm.

In radio-active changes, however, the energy is enormously greater, and hence is to be detected as before mentioned.

If we consider the whole atom is electric instead of only part, as we have considered above, a conception which is by no means artificial, since it has been proven that the mass of an electron is entirely due to its charge, we reach the interesting conclusion that on this basis the "Indestructibility of Matter" is only a corollary of the "Conservation of Energy," for if the atoms are to be considered as electrical systems, it follows that the law of the Conservation of Mass, which is essentially the same as the Indestructibility of Matter, must be approximately true, though not strictly true. It must be approximately true because no known chemical or physical change, with the exception of radio-activity, involves anything but a relatively minute liberation or absorption of energy, but the law cannot be absolutely exact, for even this minute loss of energy must involve, on the present basis, its corresponding loss of mass. Thus even the cooling of a hot body must involve a certain diminution of mass, though this, of course, is extraordinarily small, much smaller even than the loss due to most chemical reactions.

It would be interesting to search for other evidence of the existence of this vast store of energy in matter. Such evidence would involve effects which would be approximately proportional to the density. Such an effect is found in the case of the absorption of the β-rays of radium when passing through different kinds of matter. Here the absorption is proportional to the density over a very wide range. Strutt has shown that in the case of fourteen substances, whose absorption he measured, where the density varied from 0·007 in the case of sulphur dioxide to 21·5 in the case of platinum, the ratio of the absorption to the density has an average deviation of only 20 per cent.

From a physical point of view, also, it is interesting to note that, since gravitation has always been found to b strictly proportional to mass, it follows from the above that the electrical structure of matter requires that gravitation should be proportional to the energy contained in the gravitating bodies, and to this energy alone. Thus, gravitation must be considered on the present basis as existing between quantities of confined electromagnetic energy, and not between "masses" in any other sense. More knowledge bearing on the electrical theory of matter would therefore throw considerable light on the outstanding mystery of gravitation.

Summary.

We have seen that, assuming the electrical theory of matter—the theory, that is, which considers the atoms as systems composed of electric charges—it follows that the mass of a piece of matter is determined solely by the amount of electromagnetic energy which it contains and is proportional to this amount. The energy in ergs which endows 1 grm. with its mass is equal to three-fourths of the square of the velocity of light, or, in round numbers, twenty million horse-power-hours, or the energy corresponding to the work of one thousand horses working for two years. This enormous amount is by no means impossible, since the amount of energy which the radio-active substances are known to *lose* in passing to lower forms is quite appreciable in comparison.

On the electrical theory of matter it therefore follows that the chief property of matter, the property which gives us a quantitative definition, namely, its inertia or mass, is really a property of the energy stored up within the structure which defines the space relations of a piece of matter, and is not a property of the structure itself. Thus it follows that the law of the "Conservation of Mass," which we have here reason to believe is only approximate, is in reality a corollary to the law of the "Conservation of Energy," and thus this latter law and the "Indestructibility of Matter" are closely akin.

From the above it follows that on the present basis any loss of energy must involve a decrease in mass. Thus when a chemical reaction which liberates heat has taken place, when a body is cooled, or when by the process of radio-activity one substance loses energy and is transformed into another substance, there must be a decrease in the mass of the whole, and hence also a decrease in weight. In the first two cases mentioned, however, the change is too small to be detected, but in the last case the change should be appreciable, and we have a ready explanation of the irregularities which occur in the table of atomic weights.

Finally, it is pointed out that since gravity is proportional to mass it would appear that gravitation must be considered as acting between quantities of confined energy, and not between "masses" in any other sense.—*Journal of the American Chemical Society*, xxx., No. 5.

Action of Arsenic Chloride and of Arsenic on Cobalt.—F. Ducelliez.—When arsenic chloride acts on cobalt it transforms the metal into a chloride, and simultaneously an arsenide is formed. The composition of this arsenide varies according to the temperature, the limits being expressed by the formulæ $CoAs_2$ and Co_3As_2. Mixtures of arsenic and cobalt heated in hydrogen to constant weight yield the same series of arsenides. The latter compound, Co_3As_2, can also be prepared by the reduction at 900° of cobalt arsenite or arsenate by means of hydrogen. None of the cobalt arsenides are attracted by a magnet. Chlorine, oxygen, and sulphur react energetically with them; aqua regia and nitric acid dissolve them readily; hydrochloric and sulphuric acids have but little action on them; the alkalis and alkaline carbonates attack them slowly when they are fused together.—*Comptes Rendus*, cxlvii., No. 8.

SOME OBSERVATIONS ON THE ASSAY OF TELLURIDE ORES.

By GEORGE BORROWMAN.

MUCH has been written concerning the assaying of telluride gold and silver ores, yet the literature affords striking contradictions as well as statements unsupported by experimental data. However, there seems to be agreement in the opinion that tellurium is the cause of serious irregularities. Mr. A. L. Davis, in *Tech. Quart.*, vol. xii., sums up the situation as follows:—"As to the percentage of loss sustained in work, whether by scorification or crucible method, many experiments carried out upon the foregoing lines indicate to me that nothing definite can be laid down in regard to it. Every ore, every slag, every scorification, every cupel, let alone the temperature at which the assay is carried on, has some effect upon the loss, and these make too many unknown quantities to arrive at any conclusion."

It was the purpose of the writer in the work here recorded to ascertain a few facts concerning the effect of tellurium in the crucible assay for gold. The points investigated were: loss in the slag; loss in cupellation; the effect of variation in temperature of fusion.

The ore selected for the experiments was a high-grade telluride gathered from various parts of Colorado, rich in silver, gold, and tellurium. With the sulphuric acid test it gave a very strong indication of the latter element, and analysis showed the presence of 10·5 per cent chiefly as hessite and sylvanite. The gangue was silicious, consisting of quartz, felspar, and a little calcite. The ore had a reducing power of about 1·5.

The material was ground very fine, first through 120 mesh sieve, then in agate till all passed through bolting cloth. In all the work 1/10 A. T. was taken for each assay, the samples being weighed out on a quantitative balance instead of on the usual pulp scales. All fusions were made in a muffle at about 1200° excepting in Series No. 4; all the fluxes were passed through a 40-mesh sieve mixed with the ore till thoroughly homogeneous. In short, the greatest care was taken to eliminate all variations except the one studied.

Experiments were first conducted to learn the influence of tellurium in carrying gold into the slag. Mr. C. H. Fulton (*Journ. Am. Chem. Soc.*, xx., 586) records data to show that slag losses in telluride fusions are much greater than in ordinary gold ore crucible work. Messrs. Hillebrand and Allen (*Bull.* No. 253, *U.S. Geological Survey*) find losses insignificant, and commenting on the work of Mr. Fulton suggest that his losses might have been due to the use of iron nails in the fusions.

In the investigation of this point a series of assays was first made on the raw ore, using the following charge:—

Ore 1/10 A.T.	
Litharge	90 grms.
Silica..	10 „
Argols	2½ „
Sodium bicarbonate	30 „
Salt cover.	

Another series was then made on ore from which the tellurium had been removed. In removing it use was made of the fact that nitric acid dissolves tellurium and silver in a telluride and leaves the gold in the residue. Each sample was boiled with nitric acid (1·27) till no further action was apparent; the residue was washed and dried and fluxed under the same conditions as maintained in the first series. Enough silver was added to the de-tellurised series to replace what had been removed by the acid. The slags from each series were then carefully ground and assayed, using the following charge:—

Slag	
Litharge	30 grms.
Argols	2 „
Salt cover.	

The buttons from both the ores and slags were cupelled. The results are set down in the following tables:—

SERIES No. 1.

No.	Ore. Mgrms. gold found.	Ore. Ozs. gold per ton.	Slag. Gold found.
1.	16·14	161·4	None
2.	16·18	161·8	„
3.	16·18	161·8	„
4.	16·23	162·3	„
Average.	16·18	161·8	

SERIES No. 2.

1.	16·10	161·00	Trace
2.	16·16	161·60	None
3.	16·20	162·0	Trace
4.	16·11	161·10	None
Average.	16·14	161·4	

From the above data it is obvious that the influence of tellurium in carrying gold into the slag is very slight, if there is any; the amount recovered was in no case weighable.

The effect of tellurium in cupellation was next investigated, and incidentally the functions of litharge in the fusion. On the assumption that the tellurium in a crucible charge is oxidised by the litharge and carried into the slag, a series of charges was made up varying this constituent from 90 to 180 grms. Theoretically the amount of tellurium carried into the button as a lead alloy should be in inverse ratio to the amount of litharge in the fusion. A series of four samples was made in duplicate. The buttons of one series were dissolved in nitric acid and the tellurium determined by precipitating it in hydrochloric acid solution with sulphur dioxide. The other series was cupelled for gold. The two duplicates are considered as one series, *i.e.*, No. 3.

SERIES No. 3.

No.	PbO in charge. Grms.	Te in button. Mgrms.	Gold found. Mgrms.	Gold per ton. Ozs.
1.	90	lost	16·10	161·00
2.	120	287·5	16·24	162·24
3.	150	298·9	16·18	161·80
4.	180	176·8	16·15	161·50
Average			16·17	161·70

All of the beads of the above series were frosted, showing the presence of tellurium, notwithstanding the great excess of litharge in the fusion. It would then seem that litharge in reasonable amount is inadequate to slag off all the tellurium in this ore. The loss of gold, however, was inconsiderable, the average of the series varying but 0·01 mgrm. from the average of Series No. 1 in which the tellurium had been removed previous to fusion. These results are at variance with published statements regarding tellurium in cupellation. Messrs. T. J. Eager and W. W. Welch, however, found that no loss occurred in the presence of the element up to 10 per cent. The amounts in the above experiment are many times that. A bead from an assay, duplicate of No. 1 in the above series which was also the same as those of Series No. 2, was tested quantitatively for tellurium and 6·9 mgrms. were found; so that it may be present even in the bead in considerable amount without there being more than a negligible loss of gold.

The work was concluded with some experiments to learn the effect of variation of temperature of fusion on gold recovered and the amount of tellurium carried into the button. Some assayers recommend an extremely high temperature. Mr. R. W. Lodge, however, in his "Notes on Assaying," states that he believes high temperatures break up some tellurium compounds in the slag with a consequent alloying of the element with the lead.

A series of four was run in duplicate consisting of charges made up exactly as those of Series No. 2. The tempera-

ture was varied between 800° and 1600°. As in the previous experiment the buttons from one set were cupelled for gold, the other dissolved in nitric acid and assayed for tellurium.

SERIES No. 4.

No.	Temp. °C.	Weight of buttons.	Gold found. Mgrms.	Gold per ton. Ozs.	Tellurium found. Mgrms.
1.	800	30·5	16·10	161·00	258·2
2.	1000	32·5	16·16	161·60	150·0
3.	1250	32·8	16·20	162·00	195·6
4.	1600	32·8	16·16	161·60	215·9
Average			16·16	161·60	

The irregularities in the amounts of tellurium could not be accounted for, yet it seems improbable that the amount varies directly with the temperature. As to the gold the yield is greatest at about 1200°, though the average of the series is but slightly below that of Series No. 1.

Summary.

The foregoing data seem to warrant the conclusion that tellurium as the cause of irregularities in crucible work has been overestimated. Slag losses are no greater than in ordinary gold ores; the element may be present in the lead button in relatively large amounts with no considerable percentage of loss. The presence of tellurium in the bead does not necessarily imply a loss of gold in the cupellation, though, of course, a frosted bead would not be permissible when silver is to be determined. In high-grade tellurides when silver is to be estimated, the writer suggests a preliminary treatment with nitric acid, with subsequent precipitation of the silver as chloride, which may be dried and added with the residue from the acid treatment to the fluxes in the crucible. Variation in temperature of fusion does not seem to be of great moment, though the data above are most favourable to a temperature of about 1200°.

The average obtained from Series No. 1 is higher than that obtained in the others, showing a loss due to tellurium in fusion and cupellation, yet the variation is small, the average of No. 1 being not more than 0·24 per cent higher than the lowest average, that of Series No. 2. The members of the various series differ among themselves in some cases considerably, but perhaps not more than would be expected in any high-grade ore, owing to lack of homogeneity of sample. In the opinion of the writer, irregularities in high-grade tellurides are due more to this lack of homogeneity than to tellurium. It is conceivable that in some ores the ratio of gold to tellurium might be much less and hence the percentage of loss greater. In such cases a preliminary treatment with nitric acid to remove the tellurium would obviate the difficulty. — *Journal of the American Chemical Society*, xxx., No. 6.

A CHARACTERISTIC TEST FOR HIPPURIC ACID.

By WILLIAM M. DEAN.

HITHERTO no easily-applied, characteristic test for hippuric acid in urine has been at hand. For the reason that its detection in urine is of little or no clinical value, the lack of a convenient practical method for its detection has not been felt. On the other hand, for purely scientific purposes, the methods now in use involve inconveniences that are surprisingly great when compared with the methods available for the detection of other constituents of urine. As a matter of fact, the detection of hippuric acid has necessitated the concentration of the urine, the extraction with organic solvents, and either a melting-point determination of the residue, or its transformation into more easily recognised nitro or benzaldehyde derivative. (Spiro, *Z. Physiol. Chem.*, xxviii., 117; for quantitative methods, *vide* Wiener, *Arch. exp. Path. Pharm.*, xl., 314; Blumenthal, *Chem. Centr.*, 1900, II., 447).

The following test for hippuric acid can not only be applied directly to urine, but it yields definite results in solutions so dilute even as hundredth-normal. A few cubic centimetres of the urine in a test-tube are first treated with a quantity of sodium hypobromite solution, just sufficient to decompose the urea and to impart to the mixture a permanent yellow colour. The solution is then heated to boiling; an orange or brown-red precipitate is formed, if hippuric acid is present. When present only as a trace the solution appears smoky and faintly red in colour; when present in larger quantities the solution is rendered opaque and orange or brown-red in colour. In either case after standing for some time the solution clears up and a light, finely divided precipitate settles. It consists of white "earthy phosphates" intermixed with the amorphous orange or brown-red solid about to be described. (Application of the hypobromite test to aqueous solutions of other constituents of urine gave no colour effects. The following were tested:—Uric, benzoic, oxalic, and fatty acids, creatinine, acetone, acetacetic ester, glucose, glycogen, and leucine).

When a 0·1-N solution of hippuric acid is boiled with the sodium hypobromite solution, a deep brown-red colour is formed at once, and on standing settles to the bottom of the tube as an opaque, red, closely-adhering scale, which cannot be washed from the tube by simple decantation with water. With 0·01 N or even more dilute solutions, the red, adhering mass is easily observed when the liquid contents of the tube are removed.

It must be remembered that continued boiling decomposes the substance, thereby giving rise to phenylisocyanide and other products. For this reason the red substance is best prepared as follows. A concentrated solution of sodium hippurate is prepared and treated at ordinary temperatures with the hypobromite solution. After a few moments the mixture becomes red and after a few hours the precipitation is complete. It is free from the odour of phenylisocyanide and appears as a sticky, blood-red, semi-solid substance. It is insoluble in water but is soluble in most organic solvents. Its alcoholic solution is easily precipitated by the addition of water. Its solution in carbon bisulphide darkens and on evaporating leaves a green-black precipitate.

The substance contains carbon, hydrogen, nitrogen, and bromine. Limpricht and Uslar (*Ann.*, lxxviii., 133) heated hippuric acid to 240°—250° and obtained benzoic acid, benzonitrile, and resin. With sodium hydroxide and chlorine, Stecker (*Ann.*, lxviii., 54) and Gössmann (*Ann.*, xc., 181) obtained benzoylglycollic acid. The red substance obtained is not analogous to any of these. Its composition and the action of sodium hypobromite on benzamide, *p*-phenylenediamine, and other nitrogen and hydroxyl aromatic compounds is being investigated.— *Journal of the American Chemical Society*, xxx., No. 9.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 172).

Test Analyses.

NUMEROUS analyses were made to test the efficiency of the process above described. Nearly all of those which were made after the procedure assumed final form are reproduced in the tables. Almost all the analyses relating to the common elements alone were made before the procedures for the rare elements had been worked out, but the process

* From the *Journal of the American Chemical Society*, xxx., No. 4.

T. A. No.	114	115	116	117	118	119
Mn	10 S	2 S	1 S	500 S	500 S	0 S (*d*)
Al	10 S	2 S	1 D (*a*)	0 D (*a*)	2 S	0 D (*a*)
Cr	10 S	2 S	1 S	0 S	2 F (*c*)	0 S
Zn	10 S	2 S	1 S	0 S	2 S	0 S
Fe	10 S	2 S	1 S	0 S	2 S	500 S
Ni	10 S	2 S	1 S	0 S	2 S	0 S
Co	10 S	2 S	1 S	0 S	..	0 S

T. A. No.	120	121	122	123	124	125
Mn	500 S	500 S	100 S	1 S	..	..
Fe	0 S	1 S—	1 S	100 S	500 S	500 S
Ni	2 S	1 S	..	..	3 S	1 S—

T. A. No.	126	127	128	129	130
Co	0 S	0·5 S	150—	150—	150—
Ni	250—	250—	0 S	0·3 S	0·15 S

T. A. No.	131	132	133	134	135	136
Al	0 S (*b*)	0 S (*b*)	0 S (*b*)	0 S (*b*)	0 S (*b*)	0 S (*b*)
Cr	2 S	1 S	0 S	..	0 S	1 S
Zn (In P. 57)	2 F	4 F	10 F	20 S— (*e*)	5 F	0 S
Zn (In P. 67)	2 S	4 S	10 S	20 S	5 S	0 S
Mn	500 S	500 S	500 S	500 S	0 S	2 S
Fe	0 S	0 S	0 S	0 S	500 S	500 S
Ni	0 S	0 S	0 S	0 S	0 S	2 S

T. A. No.	137	138	139	140	141	142	143
PO_4	..	..	..	..	..	200 S	200 S
Zn (In P. 57)	5 F	5 S (*g*)	10 S— (*f*)	3 F	5 S— (*e*)	5 F (*h*)	10 F (*h*)
Zn (In P. 67)	5 S	5 S	10 —	3 —	5 —	5 —	10 S
Fe	500 S	500 S	500 S	..	..	..	..
Ni	..	..	..	250—	250—	250—	250 S
Co	..	..	..	250—	250—	250—	250 S

T. A. No.	144	145	146	147	148	149
Al	0 S (*b*)	3 S	3 S	0 S (*b*)	0 S (*b*)	0 S (*b*)
Cr	0 S	2 S	1 S	1 S	0 S	10 S
Zn (In P. 57)	0 S	5 S— (*e*)	3 F	3 S—	3 F	20 S— (*e*)
Zn (In P. 67)	0 S	5 S	3 S	3 S	3 S	20 S
PO_4	0 S	200 S	200 S	100 S	100 S	100 S
Fe	500 S	500 S	500 S	100 S	100 S	100 S
Ni	0 S	2 S	1 S	0 S	..	..
Co	0 S	2 S	1 S	0 S	100 S	100 S
Ba	0 S	2 S	1 S—	0 S	0 S	0 S
Ca	0 F (*i*)	0 F (*i*)	0 F (*i*)	4 S	0 F (*i*)	100 S
Mg	0 S	2 S	1 S	2 S	2 S	100 S

T. A. No.	150	151	152	153	154	155
Cr	2 S	..	0 S	..	2 S	1 S (*k*)
Zn (In P. 57)	3 F	5 S	0 S	1 F	..	..
Zn (In P. 67)	3 —	5 S	0 S	1 S	..	..
PO_4	100 S	100 S	..	..	..	..
Fe	100 S	100 S	0 S	..	..	..
Co	100 S	100 S	500 S	500 S	..	..
Ba	2 S	1 F	..	..	100 S	100 S

T. A. No.	156	157	158	159	160	161
Cr	0 S	0 S	2 S	1 S (*l*)	..	..
U	100 S	0 S	100 S	100 S	100 S	1 S
V	0 S	100 S	100 —	100 —	1 S	100 S

T. A. No.	162	163	164	165
Al	500 S	500 S	1 S	1 S
Be	0 S	2 S (*m*)	0 S	100 S

T. A. No.	166	167	168	168*a*	169	170
Zn	..	..	..	..	200 —	200 S
Al	200 —	200 —	..	..	..	200 S
Be	..	..	50 —	50 S	..	..
U (In *P. 58*c*—*d*)	2 { F —	3 { S— —	2 { F —	2 { S— S—	2 { S S—	2 { F S
U (In *P. 58*h*)	..	..	..	..	..	..

T. A. No.	171	172	173	174	175	176
Al	..	..	..	..	..	200—
Mn	..	..	..	..	..	200 S
Fe	500 S	500 S	0·5 S	500 S	500 S	200 S
Ti	0 S	1 S	5 S	..	..	..
Zr	0 S	1 S	..	..	..	..
Tl	0 S	..	..	1 S	0·5 S	1 S (*n*)

T. A. No. Begun at—	177 P. 52	178 P. 51	179 P. 51	180 P. 52	181 P. 51
Cr	2 S	1 S— (*p*)	1 S	1 S	2 F (*o*)
U	3 S	1 F (*q*)	1 F (*q*)	0 S	100 S
V	3 S	1 S	1 S	0 S	100 S (*x*)
Zn (In P. 57)	3 { S	1 { F	1 { S	1 { F	100 { S (*y*)
Zn (In P. 67)	D	S—	..	S—	S
Al	3 S	200 S	1 S (*r*)	0 S	0 S
Be	3 S	1 S	0·5 S	0 S	0 S
Mn	3 S	1 S	1 S	200 S	300 S
PO_4	..	..	..	320 S	..
Fe	3 S	200 S	1 S	1 S (*s*)	1 S (*t*)
Ti	3 S	1 S	0·5 S	1 S	0 S
Zr	3 S	1 S—	1 S	1 F (*x*)	0 S
Ni	3 S— (*u*)	1 S	1 S	200 S	1 F
Co	3 S— (*u*)	200 S	1 S	1 S	1 S
Ca	..	..	..	200 S	..
Ba	..	..	..	2 S	..

Notes to Tables.

(*a*) The presence of aluminium in the reagents made the result doubtful.

(*b*) A distinct precipitate of $Al(OH)_3$ was obtained in P. 53, but not more than with the reagents alone.

(*c*) Excess of HCl was added in P. 53 and the solution boiled, as a result of which the chromic acid was reduced. On analysing another mixture, and using HNO_3, a satisfactory test for chromium was obtained.

(*d*) The absence of manganese was confirmed by fusing some of the iron salt on platinum foil with Na_2CO_3 and KNO_3; no green colour was obtained.

(*e*) A small precipitate of ZnS was obtained in P. 57, which did not contain more than 1 mgrm. Zn. See C. E., P. 52, N. 11.

(*f*) A small precipitate of ZnS was obtained in P. 57, corresponding to 2 or 3 mgrms. Zn.

(*g*) In this analysis a large excess of alkali was added before the Na_2O_2 in P. 52, and the mixture was well stirred.

(*h*) This analysis was repeated except that the acid solution in P. 42 was poured slowly into an excess of hot alkali before adding Na_2O_2; a distinct test for zinc was then obtained in P. 57.

(*i*) A distinct test for calcium was obtained (except possibly in T. A., No. 148); the iron salt was found to contain about 2 mgrms. Ca with 500 mgrms. Fe.

(*k*) This analysis was repeated except that no Na_2CO_3 was added in P. 52; the test for chromium failed.

(*l*) See C. E., *P 58*b*, N. 2.

(*m*) The precipitate of $Be(OH)_2$ obtained in *P. 58*h* was large, and corresponded to 2 mgrms. Be, thus showing that much smaller amounts could be easily detected.

(*n*) The basic acetate procedure (P. 65) was used instead of P. 64; good tests for thallium were obtained both in the precipitate and in the filtrate. (Compare C. E., P. 65, N. 6).

(*o*) Chromium was lost in *P. 58*a*, owing to the presence of H_2O_2 (from the peruranate, see C. E., P. 52, N. 6, and *P. 58*b*, N. 2), and the addition of a large excess of acid.

(*p*) A slight blue coloration was obtained with H_2O_2 in *P. 58*b*, corresponding to not more than 0·5 mgrm. Cr. No lead nitrate was added.

(*q*) The directions in *P. 58*c* were faulty; too little phosphate was added to precipitate 1 mgrm. U.

(*r*) A good test for aluminium was obtained in *P. 58*g*, but the confirmatory test (P. 54) failed.

(*s*) The test for iron in P. 65 was a failure; the iron was found with the manganese in P. 62.

(*t*) Satisfactory tests for iron were obtained in P. 62 and in P. 64.

(*u*) $(NH_4)_2S$ was used instead of H_2S in precipitating the sulphides in P. 66, and some nickel was lost in the brown solution. Nickel and cobalt were also lost in P. 67 in the treatment with dilute HCl, owing to the rejection of the Na_2O_2 precipitate.

(*x*) Only a few mgrms. of vanadium were found in the filtrate from P. 51.

(*y*) About twice as much zinc was found in P. 57 as in P. 67.

(*z*) Owing to the insolubility of $Zr(OH)PO_4$ it is scarcely possible for zirconium and phosphate to occur together in the analysis of the aluminium and iron groups. The zirconium was undoubtedly lost in dissolving the precipitates obtained in P. 52—65.

for the analysis of mixtures containing only the common elements was at that time substantially the same as given above. In these tables the numbers in each vertical column show the weights in mgrms. of the various elements which the solution submitted to analysis contained. The results of the tests for each element are shown by the letters following these numbers. That the result was satisfactory is indicated by the letter S; that is, when the element was present, that the test for it, however small, was unmistakable, and therefore conclusive; and when the

element was absent, that a good blank test was obtained. When the test was very small, especially in comparison to the quantity of the element present, though still unmistakable, this is sometimes indicated by the symbol S—. When in the presence of the element the test failed, or in its absence a result was obtained that might be thought to indicate its presence, the letter F is used. When the result was doubtful or inconclusive, owing to the appearance or small size of the precipitate, this is indicated by the letter D. When the test was not tried, if the element was present a dash is used in place of a letter; or, if the element was not present, dots are inserted.

Common Elements.

Analyses Nos. 114—119 were begun at P. 51; P. 61 was introduced before P. 52.

Analyses 120—125 were begun at P. 61.

Analyses 125—127 were made by P. 69; analyses 128—130 by P. 70.

Analyses Nos. 131—143 were begun at P. 52.

Analyses Nos. 114—155 were begun at P. 52; P. 61 was omitted.

Mixtures containing the Rarer Elements.

Analyses 156—161 were begun at *P. 58*a*.

Analyses 162—165 were begun at *P. 58*f*.

Analyses 166—170 were begun at *P. 58*a*.

Analyses 171—176 were begun at P. 64; analysis 176 at P. 52.

Analyses 177—181 were made by Mr. R. D. Gale, who had had no previous experience in connection with the process. The first analysis was with a known mixture, the remaining four were with unknowns. Mr. Gale received no assistance while making an analysis (except in T. A., No. 179), but all the precipitates and solutions were preserved in order that the causes of mistakes might afterwards be investigated. The process was practically in its final form with exception of the procedures relating to the separation of chromium, uranium, and vanadium (*P. 58*b*—*e*), which were revised on account of the results of these analyses; moreover, uranium was not tested for in *P. 58*h*. Test analyses 156—161 and 166—170 were made at a later date to test these procedures in their final form.

Although all possible combinations of small and large quantities of the elements have not been investigated, the test analyses are sufficiently varied to justify general conclusions as to the reliability of the method of separation and detection of the elements.

An examination of these analyses shows that 1 mgrm. of any of the seven common elements in various combinations with one another and in the presence of the rarer elements was detected in almost all cases, even when the element was associated with a large quantity of other elements. Zinc frequently escaped detection in the analysis of the aluminium group (in P. 57), but was then always found in the analysis of the iron group (in P. 67) when tested for (see T. A. 131—153, 178, 180). One mgrm. is, however, near the limit of detectability when the elements of the iron group are present in large quantity. Two mgrms. of chromium were lost in two cases (T. A. 118, 181) owing to the reduction of the chromate; these analyses served to emphasise the necessity of added precautions, which have since been provided for in the directions. One mgrm. of iron in the presence of a large quantity of manganese would have been overlooked (in T. A. 180) if it had not been tested for in the solution of the chloric acid precipitate, as directed in P. 62. In the case of aluminium, owing to its being contained in the reagent, it was sometimes difficult (see T. A. 116, 117, 119, 131—136, 144—149) to determine with certainty whether or not 1 mgrm. was present.

It will be seen from T. A. 142—151 and 180 that the presence of much phosphate does not interfere with the detection of 1 mgrm. of any of the common elements of these groups. It does, however, cause a larger quantity of zinc to be completely precipitated by sodium peroxide (compare T. A. 141 with 142 and 143). It is shown by 144—146, 150, 151, and 180 that 2 mgrms. of barium and 1 mgrm. of magnesium are detected in the process, showing that adequate provision is made for the recovery of the alkaline earth elements when carried down with these groups, through the presence of phosphate.

The analyses with the rarer elements (T. A. 156 to 181) show that the process led to the detection of 1 mgrm. of each of these, and even of 0·5 mgrm. of beryllium and titanium in all cases except the following:—In T. A 180 zirconium was missed, owing to the simultaneous presence of much phosphate; but, even in the absence of phosphate, 1 mgrm. is not far from the limit of detectability in a complete analysis. In T. A. 166, 168, 170, and 178 uranium was missed in the usual place (in *P. 58*c* and *d*) owing to the fact that it was carried down with aluminium or beryllium in the treatment with dilute $NaHCO_3$ (*P. 58*a*), but T. A. 168 and 170 and a number of special experiments with mixtures of beryllium and uranium show that in such cases uranium can be detected in *P. 58*h*.

(To be continued).

IMPRESSIONS OF THE FIRST INTERNATIONAL FOOD CONGRESS.

By LOUDON M. DOUGLAS,
Lecturer on the Meat Industry, Edinburgh.

THE first Congress of an international character, which has just been held at Geneva, will be memorable because of the widespread interest it evoked. The idea of such a Congress has often been in the minds of those associated with food supply, but the standards vary so much in different countries that at the outset it may be admitted that the difficulty of meeting upon some common ground has so far prevented international action. The value, also, of any resolutions passed must, for the greater part, be of an entirely academic character, inasmuch as it is clear that indiscriminate voting at such a Congress on any specific resolution must be unequal, and the nation with the preponderance of representatives present must control the issues.

To be of any value, therefore, such Congresses must appoint an equal number of delegates who have power to vote, and it might be arranged to protect the interest of minorities by giving them power to co-opt representatives from other nations, so as to equalise their voting power.

The Congress, which was held at Geneva from September 8th to 12th inclusive, under the auspices of the Society of the White Cross of Geneva, owes its origin to Frenchmen, and Mr. Ruau, the French Minister of Agriculture, attended its deliberations, and also outlined his own and presumably his Government's attitude in relation to the food supply. The fact that out of the 400 odd delegates present from all countries some 250 were French, serves to illustrate the point that voting upon any resolution under such conditions could not be described in any sense of the word as representative of international opinion.

The primary object which the Society hopes to attain is described in the title, which states that it exists for the Repression of Frauds in Food and Pharmaceutical preparations. With such a *rôle* it will be admitted that it has a vast field of activity before it, but the political element must be suppressed, otherwise it is quite clear that such a society may be made simply the tool of whichever country cares to exploit it the most.

There were twenty-nine different nations represented, but some of these had only a very limited number of delegates present, and many were quite unofficial, having come on their own initiative. This was noticeable with

regard to the United Kingdom, which was not officially represented, although the Local Government Board had intimated that such a delegation would be sent.

The programme presented to the Congress was a large one, and dealt with many subjects in varying aspects, the discussions being largely made up of a mixture of science and commerce. The first day's proceedings were taken up with arriving at a definition of wine, but from the British point of view that is a subject which has only a local interest. The definition arrived at, however, may be of interest outside of wine-growing countries. Wine was defined as being generally the product of the alcoholic fermentation, complete or incomplete, of fresh grapes or of the juice of fresh grapes. The signification of the definition lies in the fact that during recent years much sophistication has taken place in wines, more especially for export; with the consequence that the demand has gone down considerably, and over-production has caused much depression in the wine districts of both France and Spain.

Vinegar was also defined as the exclusive product of the acetic fermentation of wine, but that is clearly a definition suited to France or Spain and few other countries. Other definitions of an equally comprehensive character were given for cider, perry, beer, and liqueurs.

The second day was devoted to defining milk, butter, cheese, eggs, fats used for foods, and various commodities associated with "charcuterie," which is in reality the business of pork purveying and the manufacture of pork products. With it, however, is associated preserved and other provisions.

The definition of butter proved to be the one in which most interest was taken, and the discussions regarding what pure butter really was sometimes became very animated indeed. Finally, however, it was agreed that butter was a mixture of fatty matters exclusively derived from cow's milk after fermentation, and made either from whole milk, separated fat, or a mixture of both.

The use of preservatives in butter naturally occupied much attention, and while many of the French delegates opposed it, others as effectively supported it, and it was soon apparent that a large number, inclusive of the British delegation, were strongly in favour of a definition that boric acid preservatives should certainly be allowed. Such opposition as exhibited itself seemed to be from a section which did not attempt to specify any objection from the point of view of health, but rather from the point of view that the use of preservatives favoured the manufacture, as distinguished from the farmer or the co-operative societies. No decision, however, was reached, and the whole question was postponed to a further conference, to be discussed under the head of "Manipulation."

The British section, through their spokesman Dr. Tunnicliffe, pointed out that the descriptive text in which the attitude of the various nations to this matter was given, was entirely wrong in stating that the use of preservative in the United Kingdom was prohibited, and that, on the contrary, it was allowed by the responsible Department of State up to 0·50 per cent. A further discrepancy in this definition also appeared in the absence of any reference to the allowable percentage of water. On the whole, the matter was left in a very unsatisfactory condition, and it may be assumed that at the next Congress the necessity for boric acid as a preservative, in addition to salt, will not only be recognised as desirable, but will be declared to be essential in all butters which have to be kept for any length of time beyond two days.

Cheese was defined as being the product of whole milk treated in the usual manner, but, of course, such a definition can have no weight whatever. The milk from which cheese is made varies considerably, as does also the recognised fat contents of cheese, and it will need much greater precision in definition before a clear understanding can be arrived at. Eggs, again, were only entitled to be described as fresh when not submitted to any process of preserving, and oils and other fats did not attain any more definite descriptions. Frozen meat was placed in the category of preserved meats!—a definition which will hardly be accorded general approbation—and sausages were only entitled to be described as "pure" when free from all other ingredients except meat, pork, or veal, in addition to the seasonings! They ought not to contain any greater percentage of moisture than the natural moisture of the ingredients. Of the other articles in the same class, none call for any special notice except salted meats, which were defined as being made from meats salted exclusively with commercial salt, with the addition of variable quantities of saltpetre and sugar. The inclusion of such a preservative as saltpetre may be regarded as indicating that the principle of using other preserving agents besides salt is unavoidable, and in this connection the general opinion was that it will be found impossible to inhibit the production of certain ptomaines without the use of boric acid or other similar preservative.

The manufacturers of cocoa seem to be very much divided as to what that article really is, as, judging from the discussion, the partisans of the use of alkaline carbonates maintained successfully that the addition of such chemical products did not interfere with the purity of the substance. The question is one for manufacturers, and some of these whose names are known in many countries insisted on the recognition of this principle.

As with cocoa and its cognate productions, so with tea and coffee. Purity from admixtures of all kinds was the keynote throughout.

In the departments of mineral waters, pharmaceutical preparations, and the unification of analytical methods, it would seem to be rather difficult to obtain such definitions as would be acceptable to all countries, as the practice must necessarily be governed by local tradition to a great extent, as well as the demands of the public. It is desirable from the International point of view that there should be unification of formulæ, but such a result can hardly be expected from a mixed Congress such as this, and, indeed would seem to be altogether out of its province.

On the whole, the first International Food Congress may be said to have been interesting as an opportunity for the exchange of opinions on many questions affecting the food supply, and the very inconclusiveness of the discussions would seem to demonstrate the necessity for better organisation in the future. The Congress may be looked upon as being established, and is likely to continue from year to year. Possibly it may be found practicable to invite the various national representatives to the United Kingdom, so that the British nation may also have an opportunity of witnessing in detail what precautions are considered necessary in the handling of food in other countries. It must always be remembered that we are more interested than other nations in this aspect of the question, as we are buyers of food supplies to a greater extent than any other.

It only remains to be said that the thanks of the Congress are due to the secretary, M. Fazy, and the Local Committee, for the excellent arrangements made. Thanks are also due to the University of Geneva for placing the rooms and hall of their classic institution at the disposal of the Congress. The ticket of membership carried the delegates to the Kursaal, and a fête at the Parc Eaux-vives arranged by the Committee of the Society of the White Cross of Geneva, and many other well-appointed functions.

As showing also the interest taken in the work of the Society, it may be mentioned that a princely donation of 100,000 francs was given by Madame Paul Bolo, and many others of a lesser amount have also been received. In so far as the British section is concerned, arrangements will be made in the future for a proper organisation, and permanent secretaries will be appointed to represent the manufacturers and the scientific sections of the United Kingdom, so that the next Congress, which will be held in Paris next year, will be attended by a British delegation worthy of the subject.

NOTICES OF BOOKS.

General Inorganic Chemistry. By ALEXANDER SMITH, B.Sc. (Edin.)., Ph.D. (Munich), F.R.S.E. London: George Bell and Sons. 1907.

A LOGICALLY presented treatment of inorganic chemistry is to be found in this book, which is suitable for the use of college students beginning the subject, or possibly for the pupils in the highest classes of schools. It is not by any means easy reading, and makes considerable demands upon the powers of concentration, but no parts are really beyond the comprehension of the well-prepared student. In one respect it differs from other books of the same scope, namely, the treatment of the theoretical side of the subject. Although there is, perhaps, not more theory than it is generally thought to be advisable to put before the first year student, the discussions of it are made much more prominent and detailed than usual, and descriptive and historical chemistry are treated only as illustrating the general theoretical principles of the science. The author thinks that the consultation of reference books for additional descriptive matter offers no difficulty to the ordinary student, while it is otherwise with theory, and there seems some justification for his idea that the books at the student's disposal should include one treating theory fully and a reference book of purely descriptive chemistry instead of one for general use, treating description fully, and a theoretical reference work. The book contains some exercises, largely on calculations, and the order in which the different subjects are introduced seems thoroughly satisfactory.

La Canfora Italiana. ("Italian Camphor"). By ITALO GIGLIOLI. Rome: Giovanni Bertero and Co. 1908.

THIS monograph is a reprint with considerable additions of reports which were presented to the Sixth International Congress of Applied Chemistry held in Rome in 1906. It gives an account of the history, commercial value, cultivation, and geographical distribution of *Laurus Camphora*, and special attention is paid to the possibilities of growing it successfully in Italy. Modern analytical researches on camphor and oil of camphor are described, and their extraction from the leaves of *Laurus Camphora* is fully treated. The bibliography of the subject is particularly carefully compiled, and includes books in all languages, and the references in the text to original papers are very full. A few notes are added on the preparation of celluloid with Italian camphor.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Berichte der Deutschen Chemischen Gesellschaft. Vol. xli., No. 11, 1908.

Titanium Nitride and a New Class of Titanium Compounds.—Otto Ruff and Fritz Eisner.—The titanium nitride to which Wöhler, Friedel, and Guérin ascribed the formula Ti_3N_4, and Schneider's "Nitride A," consist of the nitride of trivalent titanium, TiN, containing titanium nitrogen chloride and titanium dioxide as impurities, and when it is heated for some time to a high temperature in a current of ammonia, the chlorine and oxygen are completely removed and the pure nitride, TiN, is left behind. When liquid ammonia is added to titanium tetrachloride or tetrabromide and the product is washed with liquid ammonia, compounds are formed which contain at least one atom of halogen, or the corresponding amount of oxygen to one atom of titanium. They are transformed into the titanium nitrogen haloids, TiNCl and TiNBr, on heating, and on being more strongly heated they yield titanium nitride, titanium tetrachloride, or bromide and nitrogen. They are exceedingly sensitive towards moisture. The fact that the halogen is more strongly bound to titanium than to silicon (the tetrachloride of which is completely decomposed by ammonia) shows the more strongly positive character of titanium. It is noteworthy that titanium readily passes from the tetra to the trivalent state at a high temperature if it is combined with the less negative nitrogen instead of with chlorine or bromine.

Catalytic Reductions with Colloidal Palladium and Platinum.—C. Paal and Josef Gerum.—The authors have extended their experiments on the action of colloidal palladium and platinum to the unsaturated acids and their derivatives; *e.g.*, fumaric, maleic, and cinnamic acids, and cinnamic acid methyl ester. They have found that the acids are readily reduced by colloidal palladium to the saturated acid succinic acid. Colloidal platinum has a much feebler catalytic action, as shown by the much smaller velocity of the reaction.

Catalytic Reductions with Colloidal Palladium.—C. Paal and Karl Roth.—Oleic acid is reduced by colloidal palladium at the ordinary temperature, giving a good yield of stearic acid. Colloidal palladium reacts readily with castor oil, which is completely hydrated; with olive oil the hydration is not nearly so complete, though it takes place to a certain extent. Cod-liver oil under similar conditions is completely hydrated.

Lecture Experiments to Prepare Hydrocarbons.—James Frederick Spencer.—To prepare pentane (or isopentane) 2 to 3 grms. of amyl (or isoamyl) iodide are heated with the theoretical amount of dry magnesium until the reaction begins, when the heating is stopped. When the reaction is over the product is cooled, and water is added drop by drop, when pentane is evolved. Benzene can be prepared by the same reaction, benzyl iodide being substituted for amyl iodide. Similarly, α-bromnaphthalene yields naphthalene.

Coloured and Colourless Triphenylmethyl.—Julius Schmidlin.—The colourless crystals of triphenylmethyl dissolve to a clear colourless liquid in benzene, chloroform, and other solvents. The liquid turns yellow after a few seconds, but the colour disappears on shaking. The liquid again becomes coloured on standing, and loses its colour on shaking, and this process of coloration and decoloration can be repeated until finally the decolorised solution remains colourless on standing. This phenomenon is characteristic of all triphenylmethyl preparations, and apparently in the coloured solutions two chemical individuals exist, one colourless and the other coloured. Each form can be changed into the other, and in the solid and also in the dissolved state the two forms are in a definite state of equilibrium, which is dependent on the solvent and the temperature. The colourless triphenylmethyl is less soluble than the coloured variety, for triphenylmethyl separates from the yellow solution in colourless crystals.

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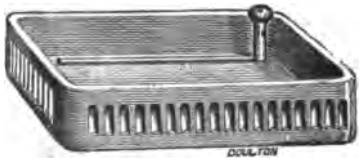

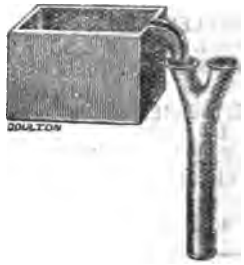

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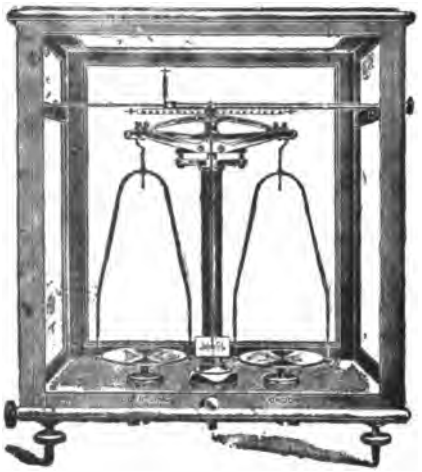

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THE CHEMICAL NEWS.

VOL. XCVIII., No. 2551.

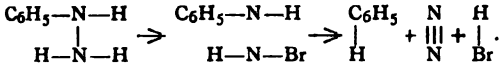
THE ACTION OF HALOGENS UPON AROMATIC HYDRAZINES.*

By F. D. CHATTAWAY, D.Sc., F.R.S.

THE actions which take place when the halogens are brought into contact with primary aromatic hydrazines are so violent, unless special precautions are taken, and such large amounts of tarry matters are formed, that they have been investigated comparatively little, and in consequence the formation of the many different substances which are produced when the conditions of interaction are altered has never been accounted for satisfactorily.

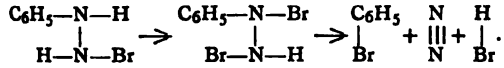
The complicated changes which occur all depend, however, upon a progressive substitution of hydrogen attached to the nitrogen, followed by a breaking down of intramolecular rearrangement of the N-substituted hydrazines thereby produced.

When a halogen acts upon an aromatic hydrazine one of the hydrogen atoms of the —NH—NH_2 group, probably one in the —NH_2 group is replaced,† a nitrogen halogen derivative being produced which at once undergoes what is in fact the typical diazo decomposition—for example :—

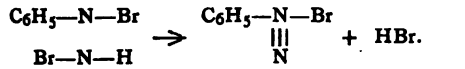


Hydroxyl ions much accelerate the rate at which this decomposition occurs, while hydrogen ions correspondingly diminish it.

When halogens therefore act upon aromatic hydrazines in presence of alkalis, hydrocarbons, the hydrides of the aromatic groups present in the hydrazines, are almost exclusively produced. When, however, an excess of halogen is employed a relatively small amount of the N-monosubstituted hydrazine undergoes further substitution before disruption can take place and a dihalogen N-substitution product is formed, which, like the monosubstitution product, is unstable in presence of alkali and breaks down similarly, a monosubstituted halogen derivative of the hydrocarbon formed in the first action being produced—for example :—

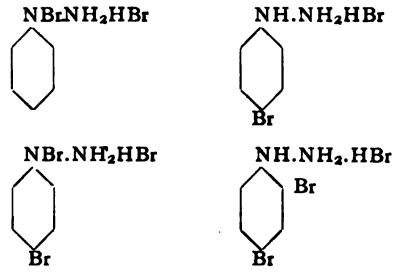


The N-monosubstituted hydrazines being far more stable in presence of acids, when these are present this replacement of a second halogen atom takes place to a much greater extent, and the whole of the hydrazine may thus be converted into the N-substituted derivative, which then may, according to conditions, undergo the diazo decomposition or yield a diazonium salt, thus :—



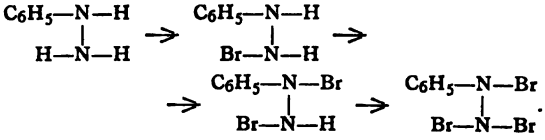
The diazonium salt may, if suitable means are adopted, be isolated (Chattaway, *Trans. Chem. Soc.*, 1908, xcviii., 852), but as the interaction of halogen and hydrazine is usually very vigorous and accompanied by considerable evolution of heat, it is generally decomposed, giving rise to tarry matters.

In presence of a considerable excess of hydrochloric or other strong acid the NH_2 group is to a large extent protected* against the action of the halogen and substitution takes place most readily in the —NH— group. The nitrogen halogen compound thus produced is of the type of the acyl amino compounds, and under the influence of the acid undergoes the intramolecular rearrangement characteristic of these compounds, whereby the halogen passes into the para or ortho position in the nucleus. Mono and disubstituted hydrazines are thus produced—for example :—

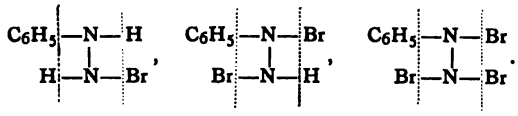


Further substitution into the remaining ortho position has not been observed, and probably cannot be effected, since the substitution of halogen so lessens the basicity of the hydrazine that the NH_2 group is attacked by preference ; indeed the yield of mono or disubstituted hydrazine is never satisfactory, diazonium salts and their decomposition products always being produced in large quantity.

The author has recently shown (*Proc. Chem. Soc.*, 1908, xxiv., 172) that when the required amounts of bromine are added to aromatic hydrazines dissolved in acetic acid, diazonium perbromides, which are thus N-tribromosubstituted hydrazines, are produced quantitatively. The replacement of hydrogen in the —$NH.NH_2$ group can thus be carried to completion—for example :—



Each of these substitution products can undergo the typical diazo decomposition quantitatively when the conditions are favourable. Thus by the action of bromine upon phenylhydrazine we can obtain either benzene or bromobenzene, nitrogen and hydrogen bromide or bromine being eliminated thus :—



Other compounds besides these simple ones may be formed—for example, from among the products of the action of bromine on phenylhydrazine, in addition to benzene and bromobenzene, *p*- and *m*-dibromobenzene and 1, 2, 4 and 1, 3, 5 tribromobenzene have been isolated, as well as azobenzene and diphenyl ; the formation of all these products can be simply explained by the theory of the reaction here put forward. It is, moreover, entirely in harmony with all that has been observed in the oxidation of the aromatic hydrazines.

* A Paper read before the British Association (Section B), Dublin Meeting, 1908.

† This, as in other similar cases, is without doubt effected by the addition of two atoms of the halogen, followed by the elimination of a molecule of halogen hydride.

* This is probably due to the addition of halogen necessary for substitution to take place being prevented by the combination of the NH_2 group with the acid.

THE RATE OF PRODUCTION OF HELIUM FROM RADIUM.

By Sir JAMES DEWAR, M.A., Sc.D., LL.D., F.R.S.

Some time ago I communicated a paper to the Society entitled "Note on the Use of the Radiometer in Observing Small Gas Pressures: Application to the Detection of the Gaseous Products produced by Radio-active Bodies" (*Roy. Soc. Proc.*, 1907, A, lxxix., 529). In the course of the experiments recorded in that paper it was shown that a pressure of the fifty millionth of an atmosphere could easily be detected by radiometer motion, and that the helium produced by radio-active processes from some 10 mgrms. of bromide of radium could be definitely detected after a few hours. This led me to desire some direct measurements of the amount of helium produced by radium, and through the kindness of the Royal Society in allowing me the use of some radium chloride belonging to them I am able to give a condensed abstract of the experimental results so far obtained.

The salt employed was the 70 mgrms. of radium chloride prepared by Dr. T. E. Thorpe, F.R.S., for his determination of the atomic weight of radium, the preparation of which is fully described in *Roy. Soc. Proc.*, A, lxxx., 298.

The apparatus used for the measurements was a McLeod gauge in the construction of which no indiarubber joints were used; the mercury reservoir being connected to an exhaust pump, while the elevation and lowering of the mercury was carried out by admitting and exhausting air in the reservoir. The air coming in contact with the mercury was purified by passage over stick-potash and phosphoric anhydride. Sealed on to the gauge was a long U-tube containing a ½ grm. of cocoanut charcoal placed in a small enlargement at the bend, the whole being arranged for liquid air or other cooling for any desired length of time. The object of the use of this cooled charcoal is to take up and condense all adventitious gases, other than hydrogen or helium, which might arise from minute leakage or otherwise be generated in the apparatus. The radium chloride was contained in a small bottle standing in a cylindrical glass bulb connected by a T-joint to the U-tube. To the other arm of the T was sealed a bulb containing about 15 grms. of cocoanut charcoal for producing a high exhaustion in the apparatus when cooled to −190° C. The whole apparatus was well exhausted by mechanical means, all the glass tubes being heated as well as the charcoal receptacles and the radium chloride. On immersing the receptacle containing the 15 grms. charcoal in liquid air for some hours, while the ½ grm. charcoal and the radium chloride were kept hot, an exhaust of 0·00015 mm. was obtained. This charcoal receptacle was now sealed off, and the small ½ grm. charcoal tube cooled in liquid air. In two hours an exhaust of 0·000054 mm. was reached.

The volume of the gauge and apparatus being approximately 200 cc., a knowledge of the pressure in the apparatus gives by a simple calculation the actual volume of gas produced measured at atmospheric pressure and the temperature of the laboratory, and thus the rate of production of helium is obtained. This, referred to the weight of radium present, gives the increment in terms of cubic millimetres of gas per grm. of radium per day.

During the first three days the growth of pressure was very small, amounting to about 0·3 cub. mm. per grm. of radium per day. This was, however, practically all produced in the first day. On then heating the radium the pressure was increased to an amount corresponding to an increment of 0·99 cub. mm. The laboratory having to be closed for a fortnight no observations were taken, and as no part of the apparatus was cooled the emanation had free play throughout. About the 350th hour after heating the radium salt the pressure had increased to a value exactly corresponding to the 0·99 cub. mm. increment observed after the first three days. This rate was, however, only kept up for the two succeeding days during which the radium was not heated. On heating the radium a further increase of pressure was obtained (corresponding to a 1·1 cub. mm. increment measured from the start), which largely disappeared on cooling the radium, and did not reappear on presently heating the radium again. From this stage throughout the next 120 hours the pressure rapidly became less, despite the heating of the radium, which only temporarily and partially restored it; and after 610 hours a lower pressure was recorded than that obtained immediately after the period of no observations, *i.e.*, after 400 hours.

The charcoal was now heated to 450° C. by boiling sulphur. Then on again cooling it with liquid air the pressure was found to have been restored by an amount equal to one-third of that lost as stated above. This, however, quickly disappeared, and apart from fluctuations caused by heating, the radium remained during the next 300 hours at about the value observed just before heating the charcoal to 450° C.

At this point the charcoal was again heated in boiling sulphur, and the previous result was repeated. In the ensuing 150 hours, however, the pressure, after falling a little, remained fairly steady, and then showed a definite and maintained increase for three days, not permanently affected by again heating the charcoal to 450° C.

The quantity of permanent gas produced up to 1100 hours corresponded to an increment of 0·417 cub. mm. per grm. of radium per day taken over the whole period. At this point the radium was sealed off, and the first experiment ended.

The large increase over the period in which the charcoal and the radium were both at ordinary temperatures may find some explanation from the unchecked action of the emanation on the charcoal, organic matter, and combined moisture possibly present on the walls of the glass tubes of the apparatus, in quantities though small yet large enough to produce the total amount of gas present which, as measured with charcoal at the ordinary temperature, corresponded to a pressure of 1 to 2 mm.

It may be noted that on the supposition of this gas being largely due to a continuous air leak, the amount of uncondensed gases of the helium type thus introduced would be infinitesimal.

Apart from the possible presence in the apparatus of organic material the radium itself might conceivably at the beginning have been contaminated with traces of organic matter, and a further experiment was decided on to which these objections could not be applied.

In the second experiment the gauge as well as the connecting tubes were well cleaned out with nitric acid, and all thoroughly dried. The radium, after the 1100 hours in which it was under high exhaustion and had been frequently heated, was certainly in a more satisfactory condition. Further, to prevent the unchecked action of the emanation throughout the apparatus, the little charcoal condenser was maintained at a degree or two below that of the boiling-point of oxygen by the use of old liquid air for a period of about six weeks. A larger quantity of charcoal was used, viz., 1 grm., the more effectively to condense out extraneous gases while leaving any helium substantially unaffected. This charcoal had been treated with chlorine at a red heat and subsequently with hydrogen.

Beyond this the conduct of the experiment followed the lines of the former one. The mercury pump exhaust was continued for several hours, and was carried to 0·002 mm. The large charcoal bulb was then cooled for several hours in liquid air while heating the 1 grm. of charcoal and the radium salt. A pressure as low as 0·00005 mm. was thus obtained when the charcoal was sealed off. On now placing the U-tube containing a small quantity of charcoal in liquid air the pressure registered was 0·000044 mm.

These conditions were maintained for five days, during which a steady growth of pressure was observed corresponding to an increment of approximately 0·3 cub. mm. per grm. of radium per day. The radium was then heated with a small Bunsen flame as before to a low red heat, when the pressure was increased by about 40 per cent. This increase showed no sign of disappearing, but during

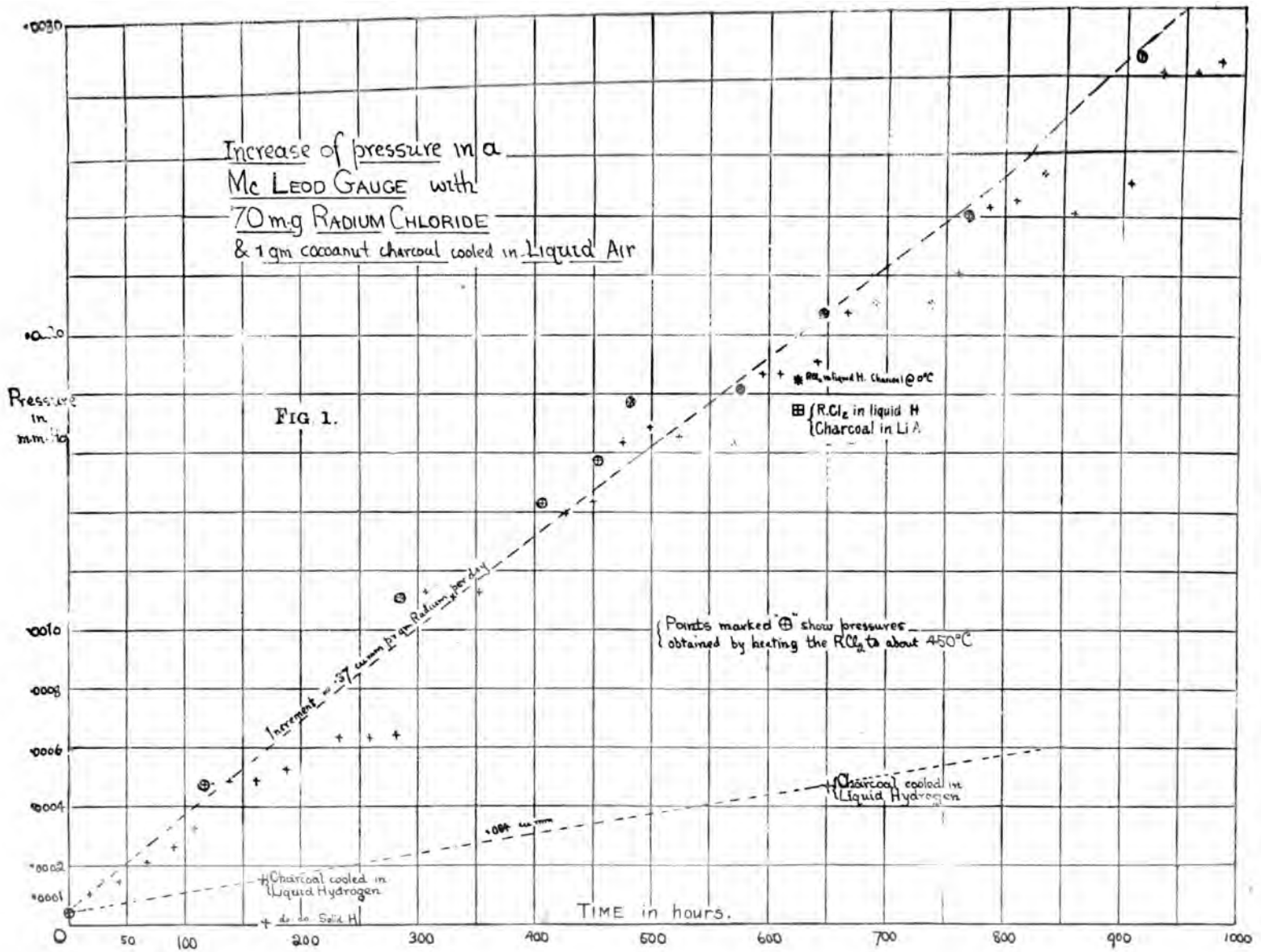

Increase of pressure in a
Mc LEOD GAUGE with
70 m.g RADIUM CHLORIDE
& 1 gm cocoanut charcoal cooled in Liquid Air
Pressure in m.m. Hg
.0030
.0020
.0010
.0008
.0006
.0004
.0002
.0001
0
50
100
200
300
400
500
600
700
800
900
1000
TIME in hours.
Increment = .37 cu mm per gm Radium per day
.084 cu mm
Points marked "⊕" show pressures obtained by heating the RCl2 to about 450°C
⊞ {R.Cl2 in liquid H / Charcoal in Li A
Charcoal cooled in Liquid Hydrogen
Charcoal cooled in Liquid Hydrogen
do. do. Solid H

FIG. 1.

the next week a decided but somewhat irregular growth of pressure was recorded. The radium was again heated. when a further increase of pressure was observed. In the succeeding five days it remained steady, only to be again increased on heating the radium. This treatment was repeated in all ten times at varying intervals during 1100 hours, and in each case the pressure rose on heating, and remained fairly steady on standing. All the observations of the second set of experiments are graphically represented in Fig. 1. A mean line is drawn through the observations taken with the radium heated, giving a steadily maintained helium increment of approximately 0·37 of a cub. mm. per grm. of radium per day.

In order to ascertain if any helium was occluded in the cooled charcoal and the surrounding glass, the latter was raised to a low red heat, while the tube containing the radium chloride was temporarily cooled in liquid air, with the object of condensing out and localising the emanation coming from the heated charcoal and preventing its access to the gauge. The temperature was maintained for an hour, and then the charcoal was allowed to cool and finally replaced in the liquid air. The radium chloride was then allowed to warm up, and was heated to near a low red for a short time. After these alterations no increase in pressure was observed, from which it may be inferred that the occlusion of the helium takes place mainly in that part of the apparatus where the radium chloride is situated.

On two occasions the charcoal was cooled in liquid hydrogen, viz., after 165 hours, and again after 650 hours. The proportionate reduction of pressure was the same in both cases, tending to show that the composition or nature of the gas remaining uncondensed by the liquid air remained the same throughout, although steadily increasing in quantity.

In reference to this last point a separate experiment was made in which pure helium under a small tension, produced by heating 0·5 grm. uranite and passing the gas produced over 1 grm. charcoal cooled in liquid air, was subjected to the action of ½ grm. of clean exhausted charcoal at the temperature of liquid air and liquid hydrogen respectively. The ratio of the two pressures so obtained was in close agreement with that observed in the radium experiment.

A further test of the purity of the gas producing the permanent pressure observed in the radium experiment with the charcoal cooled in liquid air was made by simply cooling the bulb containing the radium in liquid hydrogen, allowing the charcoal meanwhile to warm up to 0° C. If any hydrogen was present in the gas it is certain that there would be an increase of pressure recorded, since although hydrogen is partially absorbed by charcoal in liquid air, yet it would not be reduced in pressure by cooling in liquid hydrogen. On allowing the charcoal therefore to warm up, any hydrogen expelled would remain and cause an increased pressure. Inasmuch as an increase was not recorded, it can be safely assumed that no hydrogen is present, and thus the gas pressure measured consists entirely of helium.

A confirmation of this was made spectroscopically as follows:—Two tin-foil electrodes were placed round the narrow capillary measuring tube of the gauge, near the closed end. These were about 3 cm. long and about 1½ cm. apart, and were wired on with thin copper wire. The gas was compressed into this capillary space, as in taking an ordinary measure to any pressure of the order of 2 or 3 mm., while an induction discharge passed in the gas. The spectroscopic examination of this discharge revealed only the six principal helium lines, mercury, and a trace of the carbonic oxide spectrum. I have shown that the carbonic oxide spectrum always occurs in electrodeless tubes (see paper, *Roy. Soc. Proc.*, lxiv., 237, "On the Application of Liquid Hydrogen to the Production of High Vacua, together with their Spectroscopic Examination").

I am not aware of any previous direct measurements of the rate of production of helium from radium, but in a paper on "Some Properties of Radium Emanation," by A. J. Cameron and Sir William Ramsay (*Journ. Chem. Soc.*, 1907, p. 1274), the ratio of the amount of helium produced to that of the emanation was found to be 3·18, and as the amount of the emanation found by them was about 1 cub. mm. per grm. of radium per day, the resulting helium according to this experiment ought to reach about 3 cub. mm., or at least eight times the rate of production found in the above experiments. I am at a loss to explain the origin of such grave discrepancies in the measured amount of the helium produced by radium. (Prof. Rutherford, in a paper, "Experiments with Radium Emanation," *Phil. Mag.*, July, 1908, shows this result is at least ten times too great, his value being of the order 0·11 cub. mm. of emanation per day, whereas from my experiments the rate of helium production is just three times this amount). On the other hand, Prof. Rutherford, in his work entitled "Radio-active Transformations," 1906, p. 186, on the theoretical assumption that the α-particle is an atom of helium carrying twice the ionic charge, deduced from electrical measurements that the number of particles expelled per year per grm. of radium would reach 4×10^{18}, and as 1 cc. of a gas at standard temperature and pressure contains $3{\cdot}6 \times 10^{19}$ molecules, the volume of helium produced per year would amount to 0·11 cc., which is equivalent to about 0·3 of a cub. mm. per day. Considering I have found a rate of helium production of the order of 0·37 cub. mm., the agreement between experiment and the theoretical prophecy of Rutherford is almost too wonderful, substantiating as it does the accuracy of the theory of radio-active changes he has done so much to initiate and develop.

I have to express my obligations to Mr. Robert Lennox, F.C.S., and Mr. W. J. Green, B.Sc., for aid given in the conduct of these long and laborious experiments.—*Proceedings of the Royal Society*, 1908, A, lxxxi., 280.

THE FRUIT OF THE *CAULOPHYLLUM THALICTROIDES* AND *CORNUS SERICEA*.

By EDITH STOCKTON and C. G. ELDREDGE.

THE fruit of the *Caulophyllum thalictroides*, blue cohosh, was gathered at Sylvan Beach, New York, August 21, 1907. It consists of drupes with a very thin pulpy layer. The drupes were green when first gathered, but on placing them in an air-bath at 105° they quickly turned blue, and became black as the drying slowly proceeded. They possess an odour something like that of bee-bread, were about the size of small marbles, and their average weight was about 0·2 grm. There were 296 grms. available for the analysis.

The Extraction of the Sugars.

One hundred grms. of the berries were placed in a 500 cc. flask, fitted with an inverted condenser. About 200 cc. of alcohol was added, and the flask was kept for ten hours on the hot-water bath. The alcoholic extract was poured into a second flask, and the alcohol distilled off. A gummy substance remained behind. The fruit was treated in this way for about seventy days, with as many different portions of alcohol, until the extract showed no further test for sugar with Fehling's solution. The alcohol which distilled over was colourless, but a sharp penetrating ethereal odour persisted. The odour of the syrupy residue was a mixture of caramel and liquorice, the latter especially noticeable when there was an inhalation far back in the nose and throat. The syrup had a bitter taste, and resembled liquorice when first taken into the mouth. The alcoholic extract and sugar solution both gave a decided acid reaction to litmus.

The effort was made to determine the amount of sugar by using Fehling's solution, the sugar equivalent of which was known. The result was somewhat unsatisfactory, but it was afterwards noticed that the fruit originally taken had

lost 40 per cent in weight. The kind of sugar was determined by the time of osazone formation. It was thus shown to be fructose. This was confirmed by crystallising from alcohol, when the melting-point of the purified crystals was found to correspond to that of fructose.

The Oils.

The nutlets from the original berries after the alcohol extraction, as described in the foregoing, were dried in the air-bath to remove all traces of alcohol. An attempt was made to crush the nutlets in a heavy agate mortar without success. They were next transferred to a large iron mortar. They proved to be very tough. Finally, by running them through a Taylor hand ore crusher ten or twelve times, they were reduced to a coarse powder. The particles of steel from the crusher were removed with a magnet.

The crushed nutlets were placed in a 250 cc. flask to which was attached an inverted condenser, and treated with fifteen successive portions of ether on the warm water-bath. The colouring matter had been mostly removed by the alcoholic treatment, and the oil from the ether was of a clear golden-brown colour. Two kinds of oil were obtained; one of a lighter hue than the other. The darker one was very volatile, and escaped before it could be tested. The odour of the golden-brown oil resembled that of olive oil.

One hundred grms. of the original fruit were crushed and treated with ether to extract the oils. This time the oils were dark green and light green in colour. When both were warmed on the water-bath the lighter coloured seemed to disappear, although the volume remained constant. A solid substance collected on the sides of the test-tube that contained the oils. Examined under the microscope, it was needle-shaped crystals, which were in too small quantity for identification. The green oils possessed an odour like the crushed fruit, but it is difficult to describe. The golden-brown oil and the green oil were both saponified, and the saponification equivalent agreed quite closely. The two are doubtless identical. In some respects the properties would place it in the laurin group, and in other respects it would seem to belong to the olein group. About 3 grms. of oil were obtained from 100 grms. of the fruit.

The Ash.

A portion of the crushed fruit was heated in a platinum dish. The odour resembled that of burning leaves. There resulted 0·0810 grm. of ash from 2·034 grms. of the fruit. This was separated in the usual way, and found to be:—

SiO_2	5·93	per cent of the ash
Fe_2O_3	1·97	„ „
CaO	10·74	„ „
MgO	2·35	„ „

Still another portion of ash by the J. Lawrence Smith method gave:—

K_2O	36·44	per cent of the ash
Na_2O	8·69	„ „

There was also found 30·61 per cent SO_3 and 3·67 per cent P_2O_5.

Other Substances.

The syrup which was extracted by the alcohol was found to contain small quantities of citric, tartaric, and tannic acids.

The Cornus Sericea.

The fruit of the *Cornus sericea* (*Kinnikinnik*) was gathered at Sylvan Beach, New York, September 6, 1906. It was found on shrubs about 10 feet in height. The fruit was dark blue in colour, and consists of a two-celled one-seeded drupe, each one twenty-third of a grm. in weight. There were 306 grms. of the air-dried fruit on hand for the investigation.

The Pulp.

Fifty grms. of the whole berries were placed in a 500 cc. flask, which was fitted with an inverted condenser, and treated with alcohol for three or four days on a warm water-bath until all colour was removed. Eight or ten portions of alcohol were required. The extract, which was amber to brownish in colour with a slight greenish cast, was transferred to a larger flask, and the alcohol removed by distillation. The distillate carried no odour of the fruit with it, and the remaining syrup had no distinctive odour.

A portion of the berries was also treated with water, yielding an extract of a darker colour than the alcohol. Some of this solution was treated with a relatively large amount of purified bone-black for fifteen to twenty hours on the water-bath, when it lost all of its colour. This solution yielded rather unsatisfactory results, and was finally discarded. A portion, however, was evaporated to dryness on the water-bath, when there was found a black residue on the sides of the dish and a number of small crystals in the centre. The crystals were shown to contain the bases potassium and calcium, and tartaric and oxalic acids, which seemed to be united with the bases to form acid potassium tartrate, acid potassium oxalate, with some calcium oxalate. The presence of potassium was apparent whenever any of the syrup, crystals, or fruit was held in the flame. The test was also confirmed with platinic chloride.

A portion of the water solution was cleared with basic lead acetate. In addition to the substances mentioned in the foregoing, there were found tannic, gallic, and malic acids.

The Sugars.

A portion of the filtrate from the basic lead acetate was evaporated to dryness on the water-bath with constant stirring. There were weighed out 0·2 grm. portions, which were treated according to the method of Sherman and Williams (*Journ. Am. Chem. Soc.*, May, 1906). The times of the trials pointed to fructose, with portions of 0·1, 0·05, 0·01, and 0·005 grm. The osazone was filtered off and purified by repeated crystallisations from alcohol. It was found to melt at 204—205°, which is the melting-point of both dextrose and lævulose. A microscopical examination of the crystals showed their identity with the osazone of the sugar found in the urine. The sugar is deliquescent, returning to a thick syrup on exposure to the air for a short time. The sugar is neither bitter nor astringent, but resembles molasses in taste.

The Nutlets.

The berries from which the sugar had been extracted were dried in the air-bath and ground in a coffee-mill. The moderately fine powder was treated with two solvents, alcohol and ether. The apparatus used was a 250 cc. flask, fitted with an inverted condenser; the treatment, first with alcohol, then ether, lasted several days. The ether was the better solvent, and extracted a light amber-coloured oil. This was treated with bone-black, which removed all colour. The pure oil on standing partially solidified, separating into two portions, one a solid at ordinary temperatures, the other a liquid. The solid part would again liquefy on a slight rise in temperature. On placing them in a freezing mixture no change was observed. We did not succeed in saponifying them. Judging from their physical characteristics the oils seemed to be palmitin and olein.

Substances Found.

Acid potassium tartrate, acid potassium oxalate, calcium oxalate, tannic, gallic, and malic acids, fructose, palmitin, and a small amount of gum.

We desire to thank Prof. Nicholas Knight for assistance in carrying on this work.

Cornell College, Mount Vernon, Iowa,
September 25, 1908.

FIFTEENTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED DURING 1907.*

By F. W. CLARKE.

ALTHOUGH the number of new atomic weight determinations published during the year 1907 has not been large, the quality of the work done was remarkably high. Some of the determinations are of fundamental importance—as, for example, those of hydrogen, nitrogen, sulphur, potassium, and lead. Sulphur and lead are especially noteworthy, on account of their divergence from the older measurements. The data now available are as follows :—

Hydrogen.

Noyes has made complete syntheses of water in five series of determinations (*Journ. Am. Chem. Soc.*, xxix., 1718; CHEMICAL NEWS, xcvii., 56). The first series was defective, and is therefore not published by the author. For the other series very complete details are given, which would take too much space for repetition here. Noyes's corrected and final data are as follows :—

SECOND SERIES.—*Hydrogen from Sulphuric Acid, Weighed Twice.*

Weight H.	Weight O.	Weight H_2O.	At. wt. H.	
			H : O.	H : H_2O.
3·72565	29·57891	33·30408	1·00765	1·00767
3·80318	30·18400	33·98748	1·00800	1·00799
3·75873	29·83358	33·59127	1·00792	1·00795
2·96328	23·51987	26·48379	1·00792	1·00790
2·11395	—	18·89214	—	1·00795
3·53136	28·02910	31·56024	1·00791	1·00792
3·53959	28·09619	31·63554	1·00785	1·00786
		Mean	1·00787	1·00789

THIRD SERIES.—*Hydrogen from Sulphuric Acid, Oxidised by Copper Oxide.*

2·44279	19·39757	21·84042	1·00746	1·00746
2·18739	17·36305	19·55117	1·00784	1·00780
2·75129	21·84345	24·59389	1·00764	1·00768
4·00062	—	35·75073	—	1·00803
4·04057	32·07689	36·11762	1·00772	1·00772
		Mean	1·00767	1·00774

FOURTH SERIES.—*Hydrogen and Oxygen from Sulphuric Acid, combined by Palladium.*

2·27916	18·08455	20·36128	1·00823	1·00830
4·12734	32·76527	36·89043	1·00774	1·00780
4·17556	33·13449	37·30787	1·00818	1·00821
4·19346	33·27384	37·46453	1·00822	1·00831
2·30746	18·30863	20·61357	1·00825	1·00839
4·59692	36·48543	41·08162	1·00795	1·00797
4·63625	36·79354	41·42905	1·00806	1·00808
4·57274	36·28696	40·85834	1·00813	1·00817
		Mean	1·00809	1·00815

FIFTH SERIES.—*Hydrogen and Oxygen from Barium Hydroxide, combined by Palladium.*

4·61180	36·60909	41·22105	1·00779	1·00779
4·62358	36·69575	41·31647	1·00798	1·00806
4·59853	36·50484	41·10212	1·00776	1·00780
4·55832	36·17887	40·73904	1·00795	1·00790
4·20399	33·37000	37·57336	1·00782	1·00786
		Mean	1·00786	1·00788

The mean of all four series, assigned equal weight, is H = 1·00789.

The mean of the forty-eight determinations, as a single series, is H = 1·00793.

The series, however, are not of equal weight, the second and fourth being better than the others. According to Noyes, the value H = 1·00787 is the most probable value to be derived from his determinations. Morley's classical syntheses of water, re-calculated, give H = 1·00762. Combining this with Noyes's results, the average is 1·00775 which is perhaps better than the determinations of either chemist taken alone.

Silver-Nitrogen.

Richards and Forbes (*Journ. Am. Chem. Soc.*, xxix., 808; CHEMICAL NEWS, xcvi., 180), in a most careful investigation, have effected the synthesis of silver nitrate from pure silver, and so measured the ratios between Ag and $AgNO_3$. The vacuum weights are given below, together with the ratio Ag : $AgNO_3$: : 100 : x.

Weight Ag.	Weight $AgNO_3$.	Ratio.
6·14837	9·68249	157·481
4·60825	7·25706	157·480
4·97925	7·84131	157·480
9·07101	14·28503	157·480
9·13702	14·38903	157·481
9·01782	14·20123	157·480
	Mean	157·480

A small correction for a trace of water retained by the nitrate reduces this figure to 157·479, which is identical with that obtained by combining all the older data. If now, Ag = 107·930, N = 14·037; but if the recent value for N, 14·008, is correct, then Ag becomes 107·880.

Potassium.

Richards and Mueller (*Journ. Am. Chem. Soc.*, xxix., 639; CHEMICAL NEWS, xcvi., 156) have re-determined the atomic weight of potassium by analyses of the bromide. (The paper is preceded by that of Richards and Staehler on potassium chloride, which was noticed in the report of this committee for 1206; it originally appeared in the *Berichte*). First, the ratio between silver bromide and the potassium salt was measured, with the subjoined results :—

Weight KBr.	Weight AgBr.	At. wt. K.
2·19027	3·45617	39·114
4·19705	6·62285	39·113
2·06723	3·26206	39·112
2·58494	4·07889	39·115
	Mean	39·1135

Second, the ratio between potassium bromide and metallic silver was determined by the usual titration method as follows :—

Weight KBr.	Weight AgCl.	At. wt. K.
4·33730	3·93164	39·113
4·18763	3·79587	39·115
4·15849	3·76943	39·116
3·67867	3·33450	39·116
3·60484	3·26776	39·110
4·78120	4·33387	39·118
5·67997	5·14860	39·116
6·41587	5·81571	39·115
2·88134	2·61184	39·113
3·64383	3·30309	39·111
3·12757	2·83504	39·113
	Mean	39·1143

The value for K was computed with Ag = 107·93 and Br = 79·953. All weights were reduced to a vacuum standard. The final value, obtained by combining these results with those of Richards and Stahler, is K = 39·1139. Clarke, in his "Re-calculation of the Atomic Weights,"

* From the *Journal of the American Chemical Society*, xxx., No. 3.

1897, from a combination of all the older data, found K = 39·112. The two values are nearly identical.

Sulphur.

The atomic weight of sulphur has been re-determined by Richards and Jones, who employed an entirely new method (*Journ. Am. Chem. Soc.*, xxix., 826; CHEMICAL NEWS, xcvi., 201). Silver sulphate was transformed into silver chloride by heating in gaseous hydrochloric acid. The weights, reduced to a vacuum, are subjoined. In the third column the chloride formed from 100 parts of sulphate is given :—

Weight Ag_2SO_4.	Weight AgCl.	Ratio.
5·21962	4·79859	91·934
5·27924	4·85330	91·932
5·08853	4·67810	91·934
5·36381	4·93118	91·934
5·16313	4·74668	91·934
5·08383	4·67374	91·933
5·13372	4·71946	91·931
5·16148	4·74490	91·929
5·19919	4·77992	91·936
5·37436	4·94088	91·934
	Mean	91·933

From this ratio, if Ag = 107·930 and Cl 35·473, S = 32·113.

If Ag = 107·88 and Cl = 35·457, then S = 32·069.

Incidentally, the authors discuss the older values for sulphur, and especially those derived from gaseous densities. The latter they regard as subject to serious errors.

Lead.

In a preliminary paper, Baxter and Wilson give the results of their analyses of lead chloride (*Proc. Amer. Acad.*, xliii., 365). The data, with vacuum weights, are as follows :—

Weight $PbCl_2$.	Weight Ag.	Weight AgCl.	Atomic weight.	
			Ag ratio.	AgCl ratio.
4·67691	3·62987	4·82273	207·179	207·188
3·67705	2·85375	—	207·189	—
4·14110	3·21408	4·27016	207·173	207·192
4·56988	3·54672	—	207·185	—
5·12287	3·97568	5·28272	207·201	207·181
3·85844	2·99456	3·97949	207·186	207·136
4·67244	3·62628	—	207·189	—
3·10317	2·40837	3·19909	207·188	207·261
4·29613	3·33407	4·42982	207·202	207·204
		Mean	207·188	207·193

The mean of both series is Pb = 207·190 (see Note), when Ag = 107·93 and Cl = 35·473. This value is much higher than the usually accepted 206·9.

NOTE.—With Ag = 107·88, this becomes 207·090 (see *Journ. Am. Chem. Soc.*, xxx., 194).—Ed. *J. A. C. S.*

Palladium.

The determinations of Woernle (*Sitzungsb. Phys. Med. Soz. Erlangen*, xxxviii., 296) were based upon analyses of palladosamine chloride, $Pd(NH_3Cl)_2$. Two reductions in hydrogen gave the following results :—

Weight chloride.	Weight Pd.	Atomic weight Pd.
2·94682	1·48493	106·68
1·83140	0·92296	106·70

Five electrolytic determinations yielded the subjoined figures :—

Weight chloride.	Weight Pd.	Atomic weight Pd.
1·02683	0·51749	106·71
1·22435	0·61708	106·72
1·46735	0·73944	106·69
0·59796	0·30139	106·73
2·64584	1·33329	106·69
	Mean	106·708

All weights were reduced to vacuum. The antecedent values for H, N, and Cl are not stated.

Nickel and Cobalt.

Barkla and Sadler, studying the absorbability of secondary Röntgen radiations from various metals, which is an atomic function, find regularities to which nickel is an exception (*Phil. Mag.*, [6], xiv., 408; Preliminary Notice in *Nature*, Feb. 14, 1907; in *Nature* for April 4, Hackett questions the conclusions stated by Barkla). If, however, nickel be given a higher atomic weight than that now assigned to it, the anomalies disappear. Ten measurements of absorbability, compared with the absorbability of rays from other metals, gave, by interpolation, values for Ni between 61·15 and 61·6. These figures can hardly be assigned much weight in comparison with the excellent and more direct chemical determinations.

Parker and Sexton (*Nature*, August 1, 1907), in a brief note, announce that fifteen electrolytic comparisons of cobalt with silver give, in mean, Co = 57·7. The details of this investigation are yet to appear.

Indium.

Mathers (*Journ. Am. Chem. Soc.*, xxix., 485), in determining the atomic weight of indium, employed two methods. First, the ratio between $InCl_3$ and 3AgCl was measured gravimetrically, with the following results :—

Weight $InCl_3$.	Weight AgCl.	Atomic weight In.
2·1156	4·11421	114·80
4·95920	9·64176	114·85
1·98175	3·85125	114·95
5·54540	10·77904	114·90
1·46361	2·84557	114·86
4·08602	7·94054	114·96
	Mean	114·88

Secondly, similar analyses were made of indium tribromide, as follows :—

Weight $InBr_3$.	Weight AgBr.	Atomic weight In.
2·73494	4·34550	114·89
7·69880	12·23341	114·86
6·27450	9·96917	114·89
5·36642	8·52741	114·85
5·16112	8·20128	114·85
4·98336	7·92009	114·81
	Mean	114·86

The calculations were made with Ag = 107·93, Cl = 35·473, and Br = 79·953. The author favours In = 114·9 as the value to be accepted.

(To be continued).

Physical Society.—A Meeting of the Society will be held on Friday, Oct. 23rd, at the National Physical Laboratory, Bushy House, Teddington, by kind invitation of Dr. R. T. Glazebrook, F.R.S., Director of the Laboratory. It is proposed to show a series of demonstrations of work in progress in the Laboratory, which will be on view from 3.30 p.m. onwards. To give some idea of the probable number of visitors, the Director would be glad if Fellows proposing to attend or to bring visitors would intimate their intention to him by post-card, as far as they conveniently can, by Monday, Oct. 19th. He hopes, however, that no one will be deterred from coming through his having been unable to comply with this request.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 184).

Confirmatory Experiments and References.

G. D., Section 1: Precipitation of Zinc with Chromium on adding NH_4OH.—Ten mgrms. Zn as $ZnCl_2$ and 100 mgrms. Cr as $KCr(SO_4)_2$ were dissolved in 4 cc. HCl (1·12) and 96 cc. water; 10 cc. NH_4OH (0·96) were added a little at a time to the cold mixture (giving an excess of about 3 cc. NH_4OH); after three or four minutes the precipitate was filtered off, the filtrate, which had a very faint pink colour, was heated to boiling, and the precipitate that separated (estimated to contain 2 or 3 mgrms. Cr) was filtered off. H_2S was finally passed into the alkaline filtrate; no precipitate separated, thus showing that the zinc had been completely carried down with the $Cr(OH)_3$. The precipitates were united and dissolved in HCl, and the solution treated by P. 52 to 57; the zinc was precipitated as ZnS in P. 57.

For the preparation and properties of $ZnCr_2O_4$, see Chancel (*Comptes Rendus*, 1856, xliii., 927), Viard (*Bull. Soc. Chim.*, 1889, [3], ii., 331).

Action of NH_4OH *and* $(NH_4)_2S$ *on Nickel Solutions, and Precipitation of* NiS *on boiling the Brown Solution.*—See C. E., P. 51, N. 12.

The Test for Vanadium with H_2S.—See C. E., P. 51*a*, N. 1.

The Slow Reduction of Vanadic Acid to Hypovanadic Acid by H_2S.—Fifty mgrms. V as H_3VO_4 were dissolved in 4 cc. HCl and 100 cc. water; the mixture was saturated with H_2S in the cold. After about ten minutes the mixture was filtered several times to remove the sulphur that had separated, and the blue filtrate was heated to boiling; a large precipitate of sulphur separated, showing that reduction was more rapid at higher temperatures. The mixture was filtered, and the hot filtrate again saturated with H_2S; a large precipitate of sulphur separated. After ten minutes the mixture was boiled, filtered, and again saturated with H_2S; sulphur separated, showing that the reduction was still incomplete. The treatment of the hot solution with H_2S was repeated, the sulphur filtered off, the mixture was boiled in a casserole to expel H_2S, and 1 grm. KI was added; no iodine was liberated, showing that the reduction to hypovanadic acid was complete. The first experiments were repeated with 3 cc. HNO_3 (1·20) in 100 cc.; the results were the same.

Reduction of Vanadic Acid to Hypovanadic Acid by HCl (1·20).—See Gooch and Curtis (*Am. Journ. Sci.*, 1904, [4], xvii., 41). The reaction is slow in the cold but rapid on heating. When a moderate amount of vanadium is present, the reduction is not absolutely complete unless a more concentrated HCl solution is used than that obtained on evaporating. Compare C. E., *P. 58*d*, N. 1.

Action of NH_4OH *on a Hypovanadate.*—100 mgrms. V as Na_3VO_4 were treated with H_2S by P. 21 to reduce it to the hypovanadic state; the solution was evaporated to about 40 cc., and made alkaline with NH_4OH; a dirty grey precipitate separated. Fifty mgrms. V as Na_3VO_4 were boiled with concentrated HCl (1·20) to reduce the vanadium; the mixture was diluted to 100 cc., and made alkaline with NH_4OH; a small grey precipitate, with a greenish shade, separated. Several cc. NH_4OH were added; the precipitate dissolved.

Precipitation of Vanadium in P. 51 or by NH_4OH *alone when Iron or Zinc is present.*—500 mgrms. Fe as $FeSO_4$ and 100 mgrms. V as Na_3VO_4 were treated with H_2S by P. 21, evaporated to 40 cc., and treated by P. 51; the filtrate was tested for vanadium by *P. 51*a*; less than 1 mgrm. V was found. The experiment was repeated with 500 mgrms. Fe and 10 mgrms. V; no vanadium was found in the filtrate.

500 mgrms. Fe as $FeCl_3$ and 100 mgrms. V as Na_3VO_4 were dissolved in 40 cc. containing 4 cc. HCl (1·12), the solution was made alkaline with NH_4OH, and, without adding $(NH_4)_2S$, the mixture was filtered, and the filtrate was tested for vanadium by *P. 51*a*; only a trace of vanadium was found. The experiment was repeated with 500 mgrms. Fe as $FeSO_4$ and 100 mgrms. V as Na_3VO_4; the mixture was allowed to stand, and shaken frequently in order that the iron might be completely precipitated; no vanadium was found in the filtrate. Fifty mgrms. Fe as $FeCl_3$, 15 mgrms. V as hypovanadic acid, and several grms. NH_4Cl were dissolved in a little HCl and 100 cc. water; the solution was made alkaline with NH_4OH, and the filtrate was evaporated to dryness, ignited, dissolved in a little NCl, and tested for vanadium by P. 51*a*; only a trace of vanadium was found. Therefore vanadium is almost completely carried down when a three- to fivefold excess of iron is present.

300 mgrms. Zn as $ZnCl_2$ and 10 mgrms. V as H_3VO_4 were treated with H_2S by P. 21, evaporated to 30 cc., and treated by P. 51; the filtrate was tested for vanadium by *P. 51*a*; 3 or 4 mgrms. V were found. The ZnS precipitate was analysed by the regular procedure; the remainder of the vanadium was found.

Reduction of Vanadic Acid by HI *and its subsequent behaviour with the Group Reagents.*—According to Gooch and Curtis (*Am. Journ. Sci.*, 1904, [4], xvii., 45), the first stage of the reduction, that to V_2O_4, took place rapidly in a dilute HI solution, but the second stage, that to V_2O_3, takes place slowly, and only in hot concentrated solutions. When a mixture containing 60 mgrms. V_2O_3, 1 grm. KI, and an excess of HCl was evaporated to 2 cc., about 97 per cent of the vanadium was reduced to a salt corresponding to V_2O_3.

In our experiments 1 mgrm. V as Na_3VO_4 was dissolved in 15 cc. HCl (1·20) in a 50 cc. round bottom flask; 1 grm. NH_4I was added, and the mixture was evaporated carefully to 2 or 3 cc., a capillary ebullator tube being used to prevent bumping. The mixture was diluted with 10 cc. water, and NH_4OH (0·96) was added until the solution after shaking smelled distinctly of it; a dark green precipitate formed. A few drops colourless ammonium sulphide were added; the precipitate did not dissolve. It was filtered off, and dissolved in a little HNO_3; a large excess (5 to 10 cc.) of NH_4OH (0·90) was added, and the mixture saturated with H_2S; the solution became red, showing the presence of vanadium. The filtrate from the NH_4OH precipitate was tested for vanadium in the same way; only a very small amount was found. The experiment was repeated, except that the evaporated mixture was diluted to 20 cc.; more vanadium was found in the filtrate than in the precipitate. The experiment was repeated, the mixture being diluted to 40 cc.; all the vanadium was found in the filtrate.

The experiment was repeated several times with 100 mgrms. V, the mixture being diluted to 20 cc. after the evaporation; on adding NH_4OH a dark coloured (brown) precipitate separated, but it dissolved completely or in large part on adding a few drops of colourless $(NH_4)_2S$.

The experiment was repeated with 50 mgrms. V; after the first evaporation 15 cc. HCl (1·20) and 1 grm. NH_4I were added, and the evaporation repeated; the mixture was diluted to 50 cc.; a large dark greenish coloured precipitate separated on the addition of NH_4OH, and it did not dissolve on adding several drops $(NH_4)_2S$. The filtrate was evaporated nearly to dryness; an excess of 5 cc NH_4OH (0·90) was added, and the mixture saturated with H_2S; not more than 0·5 mgrm. V was found in the filtrate. The last experiment was repeated with 5 and with 10 mgrms. V; in the experiment with 5 mgrms. only a very slight precipitate was obtained on adding NH_4OH, and it quickly re-dissolved; with 10 mgrms. a precipitate

* From the *Journal of the American Chemical Society*, xxx., No. 4.

was obtained, but about half the vanadium was found in the filtrate. Therefore in a volume of 50 cc. the precipitation is fairly satisfactory for large amounts of vanadium, but not for small amounts. The above results were not perfectly reproducible, but each of them was obtained several times.

A number of attempts were made to reduce 50 to 100 mgrms. V as Na_3VO_4 by evaporating as described above with 10 to 15 cc. pure concentrated HI, and to precipitate the vanadium with NH_4OH after diluting to 40 or 50 cc.; but the precipitation was much less complete than after the reduction with NH_4I and HCl, probably owing to the larger proportion of iodine found to be retained in the solution in the former case. The experiments were repeated with HI that had been exposed to the air, and contained much iodine; the precipitation was still less complete.

Rate of Oxidation of Trivalent Vanadium in the presence of NH_4OH *and of* $(NH_4)_2S$.—Fifty mgrms. V was reduced twice by evaporation with 15 cc. HCl (1·20), and 1 grm. NH_4I to 2 or 3 cc.; the residue was diluted to 50 cc., and made alkaline with NH_4OH. The mixture was divided into two parts; one was filtered at once, and the second after half-an-hour; both filtrates were tested for vanadium by *P 51*a*; a very poor test was obtained in the first filtrate, and a good one in the second. The experiment was repeated, except that the second portion was filtered after two hours; the first filtrate gave a slight test for vanadium; the second contained 2 to 4 mgrms. V. Therefore the oxidation takes place slowly in the presence of NH_4OH.

The experiments were repeated, except that several drops colourless $(NH_4)_2S$ were also added; the filtrates obtained at once, after half-an-hour, and after two hours gave only slight tests for vanadium. Therefore the addition of a little $(NH_4)_2S$ does not cause the vanadium hydroxide to dissolve, and the rate of oxidation is slower in the presence of $(NH_4)_2S$ than in that of NH_4OH alone.

The oxidation, by the oxygen of the air of the trivalent vanadium after dilution, is therefore much too slow to account for the abnormal results, such as the nearly complete precipitation of 50 to 100 mgrms. V in one experiment, and the non-precipitation in another performed under apparently the same condition.

G. D., Section 2: Action of HCl *on* NiS *or* CoS.—Herz (*Zeit. Anorg. Chem.*, 1901, xxvii., 390, and xxviii., 343) treated sulphides which had been kept on filters for several days after precipitation with 0·5 normal HCl (1 vol. HCl, 1·12, and 12 vols. water), and found that the evolution of H_2S could not be detected by odour, or by action on paper moistened with a lead salt. On the other hand, he found H_2S to be evolved fairly rapidly by the action of 0·5 normal HCl on freshly precipitated sulphides. He concludes that there are two alloptropic forms of the sulphides, one that is soluble in 0·5 normal HCl, and one that is not. As Glixelli (*Zeit. Anorg. Chem.*, 1907, lv,, 297) has pointed out, this evidence of decreased solubility is not very conclusive, for the reaction with the old sulphides may simply be much slower. The following experiments show that NiS, at any rate while freshly precipitated, is not a mixture of two sulphides differing markedly in solubility.

200 mgrms. Ni as NiS, precipitated in the cold by passing H_2S into NH_4OH solution, was digested in the cold for a known time with about 30 cc. dilute HCl (1 vol. HCl, 1·12, and 5 vols. water). The HCl was first saturated with H_2S, and H_2S was passed through the mixture in a small flask during the treatment. The mixture was filtered, and the experiment repeated several times with the NiS residue. The amount of nickel dissolved in each treatment was estimated by adding NH_4OH, and precipitating the nickel as NiS by passing in H_2S. In the first treatment of fifteen minutes about 20 mgrms. Ni dissolved; in each of the next four treatments of fifteen minutes each, steadily decreasing amounts of Ni dissolved, and in the fifth treatment the amount was estimated to be 10 mgrms. Ni. The sixth and seventh treatments were each for half-an-hour; somewhat more nickel dissolved than in the fifth treatment of fifteen minutes. The eighth treatment was for four hours, nearly as much dissolved as in the first treatment (say, 18 mgrms.). Over-night, without passing the current of H_2S, about 20 mgrms. Ni dissolved. In a last treatment for six hours, 25 or 30 mgrms. dissolved. The final residue contained only about 30 mgrms. Ni. A similar series of experiments was performed, except that the HCl was not saturated with H_2S, and no H_2S was passed through the mixture; the results were similar except that the NiS dissolved more slowly owing to the fact that the mixture was very little stirred.

100 mgrms. Ni as NiS (precipitated by H_2S in NH_4OH solution) were digested with 30 cc. dilute HCl (1 vol. HCl, 1·12; and 3 vols. of water), for five minutes; about 5 mgrms. Ni dissolved. The experiment was repeated with HCl (1·12); the amount of Ni that dissolved was not much greater than in the preceding experiment. The last experiment was repeated with 1 mgrm. Ni; only a small proportion of the nickel dissolved.

Action of HCl *on* NiS *when other Sulphides are also present.*—500 mgrms. Fe as $FeSO_4$ and 5 mgrms. Ni as $NiSO_4$ were dissolved in 50 cc. water containing 2 cc. H_2SO_4 (1·84), and the solution treated at about 40° with NH_4OH and $(NH_4)_2S$ as described in P. 51; the filtrate was clear and light coloured, showing that it contained no nickel. The precipitate was dried by suction, and treated in a casserole with 30 cc. cold dilute HCl (1 vol. HCl, 1·12; and 5 vols. water), with frequent stirring, for five minutes; only a very small black residue remained, which was found to contain about 0·5 mgrm. Fe but no nickel. The experiment was repeated, except that ferric chloride was used instead of ferrous sulphate; the residue contained 1 or 2 mgrms. Fe and a mere trace of nickel. Both experiments were repeated, except that the sulphides were precipitated at about 90° by passing H_2S into NH_4OH solutions. In the experiment with ferrous iron the residue insoluble in dilute HCl contained nearly 1 mgrm. Fe and less than 1 mgrm. Ni; in the experiment with ferric iron the residue contained about 1 mgrm. Fe and nearly all the nickel. The last experiment was repeated with 500 mgrms. Fe as $FeCl_3$ and 2 mgrms. Ni as $NiSO_4$; the residue insoluble in cold dilute HCl contained a little iron and nearly all the nickel. The first of the above experiments was repeated with 500 mgrms. Fe as $FeSO_4$ and 10 mgrms. Ni as $NiSO_4$; the residue insoluble in dilute HCl, and also the filtrate were tested for iron and nickel; the residue contained about 1 mgrm. Fe and 5 mgrms. Ni; the HCl solution contained also about 5 mgrms. Ni. The experiment was repeated, except that the sulphides were precipitated at about 90° by passing H_2S into an NH_4OH solution; the residue insoluble in dilute HCl contained about 0·5 mgrm. Fe and 3 or 4 mgrms. Ni.

500 mgrms. Mn as $MnSO_4$ and 5 mgrms. Ni as $NiSO_4$ were dissolved in 2 cc. H_2SO_4 (1·84) and 50 cc. water, and the solution was treated with NH_4OH and $(NH_4)_2S$ as described in P. 51; the filtrate was clear, showing the absence of nickel. The precipitate was digested with 30 cc. cold dilute HCl (1 vol. HCl, 1·12; and 5 vols. water), with frequent stirring, for five minutes; the small dark coloured residue on analysis was estimated to contain 3 or 4 mgrms. Ni; the HCl solution contained only about 0·5 mgrm. Ni.

The experiment was repeated with 500 mgrms. Zn as $ZnSO_4$ and 2 mgrms. Ni as $NiSO_4$; after ten minutes' treatment with the dilute HCl a very large residue remained; the HCl solution contained only about 100 mgrms. Zn, and no Ni. The second treatment with HCl lasted over-night: about 300 mgrms. Zn and no Ni dissolved. The third treatment lasted two days; the residue was small and dark coloured, and contained over 1 mgrm. Ni and little or no Zn; the HCl solution contained about 100 mgrms. Zn and a little Ni. The experiment was repeated with HCl (1·12); a black residue remained.

These experiments show that small amounts of NiS may be dissolved completely when distributed throughout a

large precipitate of iron sulphide, but that they are not readily dissolved in the presence of ZnS or MnS. They also show that ZnS dissolves slowly in dilute HCl just as NiS does.

(To be continued).

OBITUARY.

MR. JOHN TREVOR CART.

WE regret to have to record the death of Mr. John Trevor Cart. Mr. Cart entered the School of the Pharmaceutical Society in October, 1902, as Bell Scholar—the chief distinction open to pharmaceutical students after the completion of their apprenticeship. He had served for three years in a pharmacy at Shrewsbury, and, as he had passed the "Minor" or qualifying examination of the Society before entering the School, he was granted special permission to pursue advanced studies, which led successively to his passing the "Major" examination of the Society in April, 1903; to his appointment as Demonstrator in Chemistry in October, 1903, and to his selection as Demonstrator in Pharmaceutics in April, 1904—a post which he held until he took up an appointment with the firm of Hopkin and Williams in July, 1905. There he gained additional experience in analytical and manufacturing chemistry under the supervision of Mr. Edmund White, leaving in 1906 for Newcastle-on-Tyne, where he was appointed chemist to Messrs. C. A. Parsons and Co., to carry on chemical engineering research work, and to deal with such chemical questions as arise in the carrying on of engineering and electrical business generally. He soon formed close friends with his colleagues and managers, who were attracted by his unselfish and enthusiastic nature, and his knowledge in the domain of chemistry. His work while at Newcastle threw him much into contact with Mr. Charles A. Parsons and others, who held him in their highest regard. To all who had the privilege of his acquaintance and friendship, his sudden and untimely death has come as a great shock and sorrow.

Mr. Cart was a B.Sc. of London University, an Associate of the Institute of Chemistry, and a member of the Northern Scientific Club.

Prof. W. Palmer Wynne, F.R.S., of Sheffield, says:—"During my career as a teacher I have known few more brilliant workers among my students, and none of a more amiable and unselfish disposition."

NOTICES OF BOOKS.

The Analysis of Ashes and Alloys. By L. PARRY, A.R.S.M. London: *The Mining Journal.* 1908.

THE notes on various analyses in this book will serve to recall to the chemist who is thoroughly conversant with the theory and practice of assaying details with which he cannot burden his memory, and will also give him some suggestions which he may find of value as to the relative accuracy or rapidity of different methods. At the same time the student who attempts to use it as a laboratory guide during his course of analysis will find it maddening. Details are often slurred over or even altogether omitted; many important points are left to the judgment or intelligence of the worker when he most needs some guide, and even methods of great value are left out or references to other works are given for them. However, regarded as merely outlined notes the book has good points. The directions are exceedingly concise, and the substances treated are numerous. Section I. describes the assay of the commonest metals in alloys, &c., care being taken to weigh thoroughly the merits of as many different methods as is practicable. In Section II. the analyses of ashes and slags is treated, and general methods of sampling and testing alloys are described. Section III. gives a summary of miscellaneous analyses, and in addition contains some notes on standard solutions, as well as tables of useful numerical and other data.

Der Betriebs-Chemiker. ("The Works' Chemist"). By Dr. RICHARD DIERBACH. Second Edition. Berlin: Julius Springer. 1908.

THIS book comprises a collection of notes, somewhat amplified, made by the author during his experience as a works' chemist. Thus it is essentially a practical work, and meant not for the use of students but of practical men. Moreover, it does not treat more particularly of any special branch of chemical technology, but is of a general nature, discussing problems which occur in different aspects in all industries. The cost of apparatus and materials is continually referred to, and every effort has been made to make the book of real value to the chemist. This aim is on the whole well fulfilled, and the fact that the book has found some measure of general acceptance is testified by the appearance of a second edition comparatively soon after the first.

Der Dampf in der Chemischen Technik. ("Steam in Technical Chemistry"). By W. MOMBER. Halle-a-S.: Wilhelm Knapp. 1908.

WHEN the student of chemistry has just finished his technical course and is taking a post in works or factory he frequently finds that he is required to solve problems of a very different nature from those he has investigated in the laboratory, and that his practical work does not seem to have been altogether suitable training for him. The object of this and other monographs of the same series is to bridge over this gap between works and laboratory as far as possible, and give the student a firm footing with regard to the applications on a large scale of operations and methods with which he is perfectly familiar in the laboratory. The subject treated of in this volume is of great importance, for steam is widely used in industrial chemistry for drying and heating purposes, and it is a matter of difficulty to decide what to omit, what to discuss in outline, and what to treat fully. This difficulty the author has met successfully, and the book should give the chemist very material assistance in understanding the uses of steam in the works. Different methods of producing and utilising it are described in outline, and the processes of distillation, rectification, &c., are discussed with illustrations of typical apparatus.

Die Chemische Analyse. ("Chemical Analysis"). Edited by Dr. B. M. MARGOSCHES. Vols. IV., V. By Dr. Ing. A. FISCHER. Stuttgart: Ferdinand Enke. 1908.

THE rapid methods of analysis by electrolysis which have been perfected in recent years are excellently described in his book. The general theory of electrolytic analysis as applied to the estimation and separation of various substances is first treated, and a short account is given of the apparatus used and its manipulation, although for fuller details the student is advised to refer to text-books of electro-chemistry. Some points which are of particular importance in this branch of electro-chemistry (for example, the different shapes which the electrodes may be made to assume) are considered in detail in fully illustrated discussions. In the practical part, tabulated schemes show the experimental conditions under which the electrolyses are performed, full information being given as to current, voltage, temperature, duration of experiment, &c., and the results of typical analyses are also tabulated. The author's extensive experience in practical electro-chemistry enables him to give valuable advice as to choice of methods, and some hitherto unpublished experiments performed by him are described.

Quantitative Analyse durch Elektrolyse. ("Quantitative Analysis by Electrolysis"). By ALEXANDER CLASSEN. Fifth Edition. Berlin: Julius Springer. 1908.

THE prominent part taken by the author of this treatise in the working out of methods of analysis by electrolysis fully warrants the recognition of his work as a classic upon the subject, and since its first appearance twenty-six years ago it has maintained its place as the authoritative standard work of reference. Each fresh edition has included a considerable amount of new matter, but perhaps none of them has been so much extended as the fifth, which may almost be regarded as a completely new work. The more important parts, in which great alterations have been made, include the descriptions of rapid methods of estimation, the arrangements in use at the Technical High School at Aachen being fully described. Moreover, improved methods of separating and determining the halogens, the alkaline metals, and the metals of the alkaline earths are discussed, while a new section has been added on practical applications of electro-analysis in the treatment of alloys and ores.

Jahresbericht Uber die Fortschritte des Zeugdrucks und Verwandter Industrien. ("Annual Report on the Progress of Fabric Printing and Allied Industries"). Vol. I. Edited by Prof. ANTONIO SANSONE. Leipzig: Gustav Fock. 1908.

DESCRIPTIONS of improved processes and apparatus in calico printing are published in such great numbers, and are so widely scattered in different periodicals in all languages, that the chemist finds it exceedingly difficult to keep pace with the most modern developments. Hence the value of this publication will be at once recognised, and provided that it performs its work of selection judiciously it should find a ready welcome among the readers to whom it specially appeals. The reports will be published annually, and will give a review of all matters of interest relating to cotton-printing, bleaching, and dyeing. The first number contains introductory articles, and also short chapters on the manufacture of artificial colouring matters, and on the use of the indigo-red B and thioindigo-scarlet R, the last paper being illustrated by sample patterns which have been dyed according to special formulæ given in the text.

CORRESPONDENCE.

THE THERMO-CHEMISTRY OF ALUMINIUM.

To the Editor of the Chemical News.

SIR,—Some time ago I was led to surmise that in combustion the greater portion of the heat might be given out by oxygen.

I communicated to the CHEMICAL NEWS several papers containing the theoretical reasoning leading to this, and again approach the subject of thermo-chemistry with a view to its technical application.

Aluminium when used in metallurgical processes gives out great heat.

The reaction taking place is represented by Equation I.

I. $Fe_2O_3 + Al_2 = Fe_2 + Al_2O_3 +$ . . . .
II. $Al_2O_3,yH_2O = 388{\cdot}920$
III. $Fe_2O_3,yH_2O = 191{\cdot}150$

I should surmise that the data given in Equation II. and III. represent the heat given out in Equation I., the difference being 197·770 cals.

This, though merely suggested, may be of use to such metallurgical chemists as use thermite or similar preparations.—I am, &c.,

J. C. THOMLINSON.

6, Villa View, Low Fell, Gateshead.

TENDERING FOR PUBLIC APPOINTMENTS.

To the Editor of the Chemical News.

SIR,—May we ask you to be good enough to insert in the forthcoming issue of your journal the following resolution, which was unanimously passed at a special Council Meeting of the Society of Public Analysts and other Analytical Chemists, held on the 1st inst.—We are, &c.,

ALFRED CHASTON CHAPMAN,
P. A. ELLIS RICHARDS, } Hon. Secretaries.

Resolution.

The Council of the Society of Public Analysts and other Analytical Chemists has had under consideration the practice, which has been adopted recently by certain Public Bodies, of inviting applicants for the Office of Public Analysts to state the terms upon which they are prepared to accept such appointments.

The Council is of opinion that it is against the interests of the public, and degrading to the profession of Analytical Chemistry for appointments to be offered "on tender," and calls upon the Members of the Society to resist the practice.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 9, August 31, 1908.

Dissociation Temperature of Ammonia and Carbon Monoxide.—Hermann C. Woltereck.—When pure ammonia gas is passed through a glass tube which is raised to a high temperature, the first traces of dissociation are observed at 620°, and the mixture of gases issuing from the tube becomes explosive below 630°. The decomposition of ammonia by metallic iron begins at 320°, and by iron oxide at 420°. The dissociation of carbon monoxide begins between 570° and 580°; it does not occur in presence of a trace of water-vapour even at very high temperatures.

Berichte der Deutschen Chemischen Gesellschaft, Vol. xli., No. 12, 1908.

Reaction between Ammonium Persulphate and Sodium Peroxide.—R. Kempf and Ed. Oehler.—When an alkaline aqueous solution of ammonium persulphate is allowed to stand, large quantities of nitric acid are formed, the ammonia set free by the alkali being oxidised almost quantitatively at the ordinary temperature to nitric acid. But if silver peroxide is allowed to act on alkali persulphate the free ammonia is oxidised to nitrogen. Thus the stronger oxidising agent oxidises the ammonia to the lower stage of oxidation. This phenomenon can be explained by the law of successive reactions, according to which in a slow reaction the final stable reaction products are obtained, while a rapid reaction yields less stable provisional products. Thus, nitrogen is the unstable intermediate product, and nitric acid is the stable final product. Silver peroxide oxidises large quantities of ammonia in a few seconds, while sodium persulphate takes four days to produce 0·6 grm. of nitric acid. Sodium peroxide, even in the solid dry state, reacts energetically with ammonium persulphate, the reaction being accompanied by the development of much heat and light, and sometimes by an explosion. The analytical study of the reaction shows that several chemical reactions proceed simultaneously. Thus ammonia is set free, and this is oxidised to nitrogen, as shown by the equation $(NH_4)_2S_2O_8 + 2Na_2O_2 = 2Na_2SO_4 + 4H_2O + N_2$. The heat

evolved by this reaction decomposes the oxidising agent, especially the sodium peroxide, and oxygen is evolved. No explosive mixture is obtained if the sodium peroxide is replaced by caustic soda, or if instead of ammonium persulphate, ammonium sulphate or sodium persulphate is used.

Hydrogen Persulphide.—Rudolf Schenck and V. Falcke.—To prepare hydrogen persulphide potassium polysulphide solution is poured into concentrated hydrochloric acid, and the clear light yellow oil thus formed is separated from the aqueous liquid, and washed with dilute hydrochloric acid. The oil can be dried with calcium chloride, and distilled off from the drying agent in a vacuum, the vessels in which the raw sulphide is distilled having previously been carefully freed from traces of alkali. Thus a strongly refracting oil is obtained with a characteristic odour, recalling that of camphor. The substance is very volatile and also very labile, readily splitting up in presence of small quantities of alkali into sulphur and sulphuretted hydrogen. Hydrogen persulphide is dissolved by carbon disulphide, benzene, alcohol, and ether, but is readily decomposed by solvents into sulphur and sulphuretted hydrogen. The composition of the persulphide may be determined by decomposing it completely into sulphur and sulphuretted hydrogen by means of ether, and estimating the amount of each constituent. The results obtained agree very well with the formula H_2S_3, and this formula is confirmed by molecular weight determinations in which the depression of the freezing-point is measured, bromoform being used as solvent.

Bromination Experiments with Diazobenzene Perbromides.—Carl Bülow and Herm Schmachtenberg.—At a low temperature diazobenzene perbromide is readily dissolved by acetone and acetophenone. From the solution hydrobromic acid is evolved, and a crystalline mass of pure benzenediazonium bromide is formed, while at the same time the unmistakable odour of monobromacetone or acetophenone may be recognised. The yield is quantitative as expressed by the equations:—

$$C_6H_5.N_2.Br.Br_2 + CH_3.CO.CH_3 = = C_6H_5.N_2.Br + CH_3.CO.CH_2Br + HBr,$$

$$C_6H_5.N_2Br.Br_2 + CH_3.CO.C_6H_5 = = C_6H_5.N_2.Br + CH_2Br.CO.C_6H_5 + HBr.$$

The perbromide acts upon acetic acid solutions of unsaturated compounds, such as cinnamic or phenylpropiolic acids, giving the dibrom addition products:—

$$C_6H_5.N_2.Br.Br_2 + C_6H_5.CH{:}CH.COOH = = C_6H_5.N_2.Br + C_6H_5.CHBr.CHBr.COOH,$$

$$C_6H_5.N_2.Br.Br_2 + C_6H_5.C{:}C.COOH = = C_6H_5.N_2Br + C_6H_5.CBr{:}CBr.COOH.$$

With an alcoholic solution of aniline the perbromide gives chiefly the hydrobromide salt of the base, diazoamidobenzene and small quantities of amidoazobenzene and tribromphenol. Similar results are obtained with acetyl-*p*-amidobenzenediazonium bromide.

Crystallised Boron.—Heinrich Biltz.—Kühne stated that silicon and boron are obtained in the crystalline form if their oxides are burnt with aluminium and sulphur; part of the aluminium forms sulphide, and thus sufficient heat is generated to decompose the oxide with the rest of the aluminium. The boron crystals are then said to be easily isolated from the aluminium sulphide. The author has repeated Kühne's experiments, and by an improvement of the method has obtained black crystals resembling those described by Wöhler and Deville in 1857. These authors obtained, besides the black crystals, clear yellow or brown crystals containing boron, aluminium, and carbon. They regarded the two crystalline forms as essentially identical, and thought they were elementary boron, containing carbon and aluminium as impurities. The author's experiments have shown that in the black crystals a chemical compound, an aluminium boride of formula AlB_{12}, is present.

Velocity of Reaction of Bromine on Benzaldehyde.—W. Herz and Hermann Dick.—Bromine and benzaldehyde yield brombenzylbenzoate,—

$$2C_6H_5.COH + Br_2 = C_6H_5.CO.O.CHBr.C_6H_5 + HBr.$$

It is *à priori* probable that the transposition is a bimolecular reaction, according to the equations:—

$$C_6H_5.COH + Br_2 = C_6H_5.COBr + HBr,$$
$$C_6H_5COBr + C_6H_5.COH = C_6H_5.CO.O.CHBr.C_6H_5.$$

To test this hypothesis the authors have determined the constant K from the bimolecular reaction formula $\frac{1}{t(A-B)} \log \frac{B(A-x)}{A(B-x)} = K$, and have obtained results which agree very well with the theoretical value. The values of K are independent of the concentrations within certain definite limits.

MEETINGS FOR THE WEEK.

WEDNESDAY, 21st.—Microscopical, 8. "The Mouth-parts of the Nemocera and their relation to the other Families in Diptera—Corrections and Additions to the Paper published in 1904," by W. Wesché. "Resolution of Periodic Structures" and "An Auxiliary Illuminating Lens," by E. M. Nelson.

FRIDAY, 23rd.—Physical, 3.30 (at the National Physical Laboratory, Bushy House, Teddington).

THURSDAY, 22nd.—Chemical, 8.30. "Passage of Hydrogen through a Palladium Septum, and the Pressure which it Produces," by T. Tsakalotos. "Relationship of Colour and Fluorescence to Constitution—Part II., Rhodamines of Mellitic Acid," by O. Silberrad and C. S. Roy. "Constitution of the Fluorescences of Mellitic and Pyromellitic Acid," by O. Silberrad. "A New Form of Gas Burette," by A. E. Hill. "Molecular Compound of Trinitroacetaminophenol and β-Naphthol," by R. Meldola and J. G. Hay. "Reduction Products of Azoxybenzene," by L. H. Berry. "Constitution of the Salts of Phthaleins, and the Cause of Colour in the Triphenylmethane Series," by A. G. Green. "Chlorination of *p*-Nitraniline," by B. Flürscheim. "Relation between Absorption Spectra and Chemical Constitution—Part X., Unsaturated Acids of the Benzene Series," by E. C. C. Baly and K. Schaefer. "Condensations with Monochloromethyl Ether—Part I., Condensation of Monochloromethyl Ether with Ethyl Malonate and Ethyl Isopropyl Malonate," by J. L. Simonsen. "Relation between Chemical Constitution and Physiological Action in Certain Substituted Aminoalkylesters," by F. L. Pyman. "Effect of Constitution on the Optical Rotatory Power of Optically Active Nitrogen Compounds," by R. W. Everatt and H. O. Jones.

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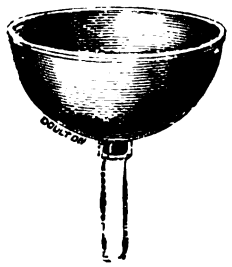

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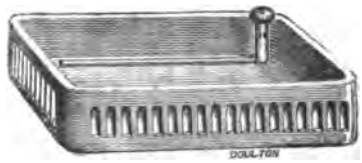

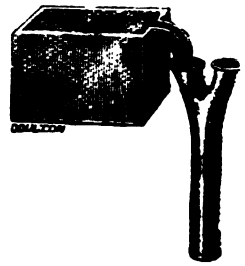

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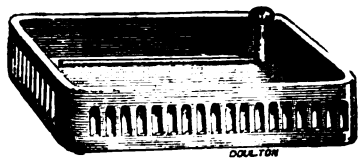

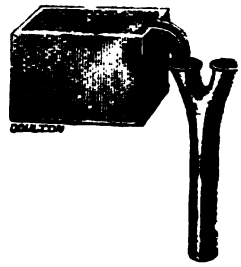

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ELECTROSCOPIC DETERMINATION OF THE RADIUM PRESENT IN SOME "TUFA" DEPOSITS FROM HOT SPRINGS, ARKANSAS.*

By HERMAN SCHLUNDT.

The Hot Springs of Arkansas are situated in the central part of the State, on the western slope of a ridge known as Hot Springs Mountain. The thermal waters issue from fifty separate vents in the old and grey hot-spring deposit, or "tufa," that covers the lower slopes of the mountain of sandstone and shale (W. H. Weed, "Geological Sketch of the Hot Springs District, Arkansas." Senate Document, 1902, vol. xx., No. 282, 79. Government Printing Office, Washington, D.C.). The total flow is estimated at 850,000 gallons per day. The thermal waters at Hot Springs correspond closely to the ordinary springs of the surrounding mountain region, save in the one characteristic of temperature. They are regarded as entirely normal springs whose waters have been heated in the course of their underground flow by steam rising through fissures in the rock, the source of the heat being a hot, igneous mass of intrusive rock underlying this portion of the State.

In 1904, Dr. Boltwood, at the direction of the Secretary of the Interior, made a thorough examination of the waters of these springs for radio-active properties. (The results are summarised in a paper published by Dr. Boltwood in the *American Journal of Science*, 1905, xx., 128). In addition, samples of gases from two of the springs were examined; the residues from two samples of water were tested, and a sample of a spring deposit of "tufa" was included in the investigation. He tested all of the waters for radio-active gases—radium emanation—and a few of them for dissolved radium salts. Two water residues and one tufa deposit were tested for radium. The results of Dr. Boltwood's investigation showed that the waters possessed radio-activity, the activity being due to the presence of radium emanation. Two cold springs on the reservation were tested and also found relatively high in activity. No indication of radium salts in solution was obtained. The deposit of tufa examined was found practically free from radium; Dr. Boltwood states that the quantity of radium present in the tufa was less than one-millionth of the quantity of radium associated with an equal weight of uranium in pitchblende; or, in other words, the radium content of the tufa has the same order of magnitude as that found in an ordinary soil sample. One of the points of special interest that the results of Dr. Boltwood's investigation presented was the existence of marked variations in the activity of the different water samples, whose mineral constituents stand in such close resemblance. No connection could be established between chemical composition of the waters and the striking differences observed in their radio-active properties.

Elaborate chemical analyses of the waters of these springs have been made by Mr. J. K. Haywood, of the U.S. Dept. of Agriculture. The published results constitute the greater portion of Senate Document No. 282, *loc. cit.*

During the past year the writer has determined the quantity of radium present in several tufa deposits from Hot Springs. The results obtained demonstrate that the quantities of radium present in some of the samples examined greatly exceed the quantity found in the particular sample tested by Dr. Boltwood. Such marked variations as he found in the quantities of radium emanation present in the water samples are likewise exhibited by the tufa samples examined.

The specimens of tufa used for the tests were obtained through the courtesy of Mr. Martin A. Eisele, Superintendent of the Reservation (Mr. Eisele resigned the superintendency, April 1, 1907). He collected them during the past winter, and expressed them to Columbia, Mo., where the writer conducted the tests herewith described in the chemical laboratory of the University.

In determining the radium content of the deposits, the method devised by the Hon. R. J. Strutt in his investigations on the "Distribution of Radium in the Crust of the Earth" (*Proc. Roy. Soc.*, 1906, lxxvii., 472) was followed in the main. In this process the material to be tested must be converted into soluble form. As the tufa is calcareous in nature, digestion with hydrochloric acid dissolves most of the material. The principal steps of the operation are as follows:—An air-dry sample of about 200 grms. was pulverised and passed through an 80-mesh sieve. It was then dried for an hour at 105° C. A suitable quantity was then weighed out and dissolved in hydrochloric acid. When the insoluble residue exceeded 2 per cent, it was filtered off and rejected. Fusion of one of these residues with mixed carbonates of sodium and potassium and subsequent solution and treatment showed that it was practically free from radium. The solution of chlorides was then stored for several weeks in a tightly-corked flask of suitable capacity to permit the accumulation of the radium emanation produced by the radium salts in the solution. After a lapse of a month, when the accumulated emanation had reached a maximum, it was separated from the solution by boiling, the expelled gases being collected in a suitable reservoir. (For a description of the apparatus employed and the experimental details, the reader is referred to a former paper by Schlundt and Moore, *Journ. Phys. Chem.*, 1905, ix., 320). The gas was then transferred to a partially exhausted air-tight electroscope supplied with a reading microscope. From the rate of fall of the charged leaf, the quantity of radium required to produce the emanation in the electroscope, *i.e.*, the radium in the sample, was finally calculated.

The testing apparatus had previously been carefully standardised, by means of a known quantity of radium emanation obtained from a sample of uraninite whose uranium content was known. This method of standardising electroscopes was first proposed by Boltwood. It is based upon the well-established fact that the radium present in a natural mineral stands in constant ratio to the uranium it contains. The operation of standardising was carried out as follows:—A few mgrms. of a standard sample of uraninite were carefully weighed out, dissolved in dilute nitric acid in an apparatus that permitted the quantitative collection of the emanation. The emanation, mixed with other gases, was then introduced into the electroscope to be standardised, and the maximum rate of fall of the charged leaf determined. From these data a factor was computed for the instrument, expressing the quantity of uranium required to produce a fall of the charged leaf corresponding to one scale division per minute of the reading microscope. The uranium values for the activities were then converted into radium values by multiplying by the ratio of radium to uranium. The recent value of this ratio, as again determined by Rutherford and Boltwood, viz., $3{\cdot}8 \times 10^{-7}$, was employed in the calculations (*Am. Journ. Sci.*, 1906, xxii., 1).

The same sample of hydrochloric acid served for all the determinations. A blank test showed no indication of radium.

The quantitative results of the radium content of the tufa samples tested are given in the following table:—The first column gives the laboratory number, the second the weight of the sample taken for the determination, the third the percentage of insoluble residue, a "small" residue meaning less than 2 per cent. The last column gives the radium found per grm. of tufa, the radium unit being 1×10^{-12} grm.

* A Paper read by title at the Twelfth General Meeting of the American Electrochemical Society at New York City, October 18, 1907.

TABLE OF RESULTS.

Laboratory number.	Weight of sample taken (grms.).	Percentage of insoluble residue.	$g \times 10^{12}$ radium per grm of tufa.
1	152	Trace	0·72
2 A	175	Small	26·7
2 B (*a*)	125	Small	26·6
3	100	Small	2·62
4 A	120	6·7	2·32
4 B	98	6·9	2·85
5 A	2	9·4	1900
5 B	1	9·4	1927
5 C	2	—	227
5 D (*a*)	?	—	1322
6	160	Small	0·73
7	120	8·1	156·0
8	100	5·5	0·56
9	50	18·0	1·18
10	140	Small	36·3
11	100	7·8	0·01 ?
12 (*b*)	100	—	$< 0·38$

(*a*) I am indebted to Prof. Moore, of Butler College for this determination.

(*b*) Sample of tufa examined by Dr. Boltwood. The value given in the table was computed from the quoted statement introduced at the beginning of this paper.

Discussion of Results.

The most interesting feature of the results obtained is represented in the marked variations in radium content exhibited by the different tufa samples. No chemical analyses of the tufas examined were undertaken, but inasmuch as the physical properties of the specimens varied considerably, differences in composition doubtless exist, notwithstanding the fact that the dissolved mineral constituents carried by the water from different springs bear a close resemblance to one another. A few of the samples had a compact crystalline structure and resembled white marble; others were porous, consisting of a framework of travertine intermingled with earthy material, in which some of the higher oxides of manganese are probably present, as considerable chlorine was set free during the digestion with hydrochloric acid. No one physical characteristic or group of physical properties could be singled out, however, as representative of the more active samples. The specimens that left only small quantities of brown oxides when treated with cold hydrochloric acid of four-normal strength were found low in radium; but the presence of a relatively large quantity of insoluble material before the temperature of digestion was raised was found not to be a general accompaniment of high activity. Finally, a careful comparison of the mineral constituents of the spring waters that deposited the tufa of high radium content with those of low values fails to furnish any clue to the marked differences found in their radium content.

As stated before, marked variations in activity also characterise the water samples. Whether a parallelism exists between the content of radium emanation in the waters and the quantity in the tufa deposits cannot be stated, as Dr. Boltwood has designated his samples by laboratory numbers. The same system was followed in this paper upon suggestion of the Superintendent of the Reservation.

It is seen that the quantities of radium in Samples 1, 6, 8, and 9 are of the same order of magnitude as the radium content of the sample of tufa that came to Dr. Boltwood for examination, its maximum radium value being the last figure in the table of results.

Samples 2A and 2B are determinations on the same composite, as are also Samples 5A and 5B. In testing specimen No. 5, the calcareous crystalline material was partially separated and tested—5C; its activity was found to be only one-ninth the value obtained for the composite of the entire sample. It therefore appears probable that the radium occurs in pockets, and that it may be associated with some one of the different constituents of the tufa. Further investigations with a view of separating the radium-bearing constituents, either mechanically or by chemical means, would be of interest.

Another question for further investigation is suggested by the results:—Is the radium, found in these tufa deposits, in radio-active equilibrium with its parent, uranium? The quantities of uranium considered here are, in most of the samples, too small to be detected by chemical analysis; but in sample No. 5 the quantity of uranium would be sufficient for an analytical attempt. With 1900×10^{-12} grms. of radium there should be associated $1900 \times 10^{-12}/3·8 \times 10^{-7}$ grms. uranium, or 0·005 grm., *i.e.*, $\frac{1}{2}$ per cent uranium. Unfortunately, in the course of the preliminary tests conducted with this interesting sample, most of it was lost, and up to the present time arrangements for securing more of the deposit have not been made.

Sample No. 11 does not properly belong in the table. This was a sample of loadstone probably deposited by hydrothermal action at Magnet Cove, Arkansas, an extinct locality of hot springs in this region.

The number of samples reported here represents but a small fraction, probably less than one-fifth of the deposits of tufa, but the results obtained are of sufficient interest to warrant a more complete investigation. An examination of the underlying sandstones and shales, and the dikes and igneous rock of the locality, would also be of interest in furnishing data as to the source of the radium in the spring deposits.

DISCUSSION.

Prof. SCHLUNDT (communicated):—Dr. Boltwood has kindly communicated the official numbers of the springs that correspond to the laboratory numbers in his paper (*loc. cit.*). This courtesy enables me to correlate the activities of the water and tufa samples. The data are summarised in the following table. The values for the activity of the water samples are expressed in grms. of uranium per litre of water, as in Dr. Boltwood's paper. The official numbers of tufa Samples 1 and 2 are in doubt, and are therefore not included in the table. Samples Nos. 8 and 9 were tufas taken from caves for which corresponding water samples were not treated.

Laboratory number of tufa sample.	Activity of tufa; $g \times 10^{-12}$ radium per grm. of tufa.	Activity of water; $g \times 10^{-4}$ uranium per litre of water.
3	2·62	5·3
4	2·65	12·9
5	1900	3·4
6	0·73	3·7
7	150	26·1
10	36·3	22·0
12	0·38	41·6

The limited number of results thus correlated—there are fifty springs—indicate that a correspondence between content or radium emanation in the waters and radium present in the tufa deposited by the waters does not exist.

SOME CHARACTERISTIC COLOUR REACTIONS PRODUCED BY SODIUM HYPOBROMITE.

By WILLIAM M. DEHN and SILAS F. SCOTT.

SINCE hippuric acid gave with sodium hypobromite a very characteristic colour reaction, it was thought that other substances also would be affected characteristically by this reagent (see *Journ. Am. Chem. Soc.*, xxx., p. 1507; CHEMICAL NEWS, vol. xcviii., p. 181). This has proved to be the case and to such an extent that sodium hypobromite alone or combined with the use of ammonia can now be considered one of the most valuable reagents for detecting and distinguishing many phenolic and nitrogenous aromatic compounds.

For the purpose of uniformity and of determining the

limits of tests and comparing their respective delicacies, solutions of the various substances were prepared in the following concentrations:—I., 1 grm. in 100 cc. of water; II., in 1000; III., in 10,000; IV., in 100,000; V., in 1,000,000.

Alcohol was added when necessary to produce complete solution; with bases, hydrochloric acid was used for the same purpose. With various concentrations of solutions quite different colour effects may often be obtained, and thus one sometimes obtains results quite different from the results described in the literature. For this reason the concentrations of solution should always be indicated, also the quantity used, method of applying the reagent, quantity of reagent, and the temperature. In the following experiments the reagent was applied, a drop at a time, to about 5 cc. of the solutions tested. When a series of colours are given they indicate the progressive effect either on standing or on adding more of the hypobromite solution. The following abbreviations are used to indicate the conditions and manner of applying tests:—

(A). Treated with sodium hypobromite at ordinary temperature.

(A'). Treated with ammonia, then with sodium hypobromite at ordinary temperature.

(B). First heated to boiling, then treated with sodium hypobromite.

(B'). Treated with ammonia, then heated and finally treated with sodium hypobromite.

Of course solutions should not be acid in reaction, for then bromine is liberated from the hypobromite. Excess of ammonia should be avoided for the reason that it destroys the hypobromite. Care should be exercised that the hypobromite is added gradually, for the reasons not only that often a series of colours may thereby be developed but that the possible destruction of colour by an excess of hypobromite may be avoided. Conditions of experiments and concentrations of solutions yielding negative results are omitted in the following descriptions. When heat and the addition of ammonia give effects that are the same as (A), only the effects of the latter are described.

Phenolic Compounds.

*Phenol.**—(B) and (B'): I., deep chrome-green; II., same; III., light sea-green. Ammonia is necessary, and heat assists this colour reaction.

Pyrocatechinol.—(A), (A'), (B), (B'): I., II., bright green followed immediately by a deep brown. (B). (B'): III., pink-brown followed by amber-brown. Most sensitive with ammonia and heat. Colour is changed to yellow by an excess of sodium hypobromite.

Resorcinol.—(A): I., bromine-brown; II., amber-brown; III., straw-yellow. (A'): I., deep brown-black; II., deep amber-green. (B): I., amber-brown; II., amber-green; III., green-amber; IV., straw-yellow. (B'): I., deep brown-black; II., deep amber-green; III., green-amber; IV., faint pink. Colour is intensified by the use of ammonia and heat; destroyed by an excess of sodium hypobromite and continued heat.

Hydroquinol—(A): I., chrome-green, changing immediately through bromine-brown to deep brown-black; II., chrome-green to deep brown; III., transient light green. (A'): I., deep brown; II., same; III., very transient light green. (B): Ammonia itself imparts a golden-amber hue; I., dark brown; II., deep amber; III., light amber. Less sensitive with ammonia and heat; an excess of sodium hypobromite discharges the colour, yielding odour resembling quinone.

To Distinguish the Dihydric Phenols.—To about 0·1 per cent solution of each add one drop of ammonia; hydroquinol gives a golden-yellow colour. Add 2—3 drops of sodium hypobromite; pyrocatechinol gives a dark brown, resorcinol gives a green, and hydroquinol gives a brown-red colour.

* A blue colour with bleaching powder is described: Lex, *Ber.*, iii., 458; Salkowski, *Zeit. Anal. Chem.*, xi., 316; *Jsb. Chem.*, 1873, 722.

Pyrogallol.—(A): I., amber, brown-red, deep black-brown; II., amber, deep brown-red; III., amber. (A'): Same as (A) but less sensitive. (B): Ammonia alone imparts an amber colour; I.—III., same as above. IV., amber.

Phloroglucinol.—(A): I., green-amber, green-brown, deep violet, deep violet-brown; II., violet, red-brown, amber. (A'): I., black-brown; II., brown-red. (B) and (B'): I., deep violet, deep brown-black; II., deep violet; III., violet. The violet colour, best formed with ammonia in a warm solution, easily distinguishes phloroglucinol from the other phenols.

Orthocresol.—(A): I., dirty chrome-green, brown, dirty brown; II., brown-amber, brown. (B): I., Paris green, deep black-green, dirty green, yellowish precipitate; II., brilliant chrome-green, grass-green, amber; III., sea-green, straw-yellow.

Metacresol.—(A): I., pink-amber, light-brown; II., amber. (B): I., blue-green, deep black-green, brown, amber, dirty white precipitate; II., brilliant emerald-green, green-amber, amber; III., light green, amber.

Paracresol.—(A): I., pink, pink-amber, light brown, dirty white precipitate; II., pink, amber; III., light yellow. (B): I., light amber, brown; II., amber.

The cresols are distinguished by the lack of formation of a green colour with the para, when treated with ammonia and sodium hypobromite; the ortho forms a dark brown, the meta a light brown, when treated with sodium hypobromite.

"Cresol."—(A): I., pink, wine-red, deep brown-red; II., amber, pink, amber, red, brown; III., light yellow. (B): I., sea-green, deep dirty green, deep brown; II., light pink-amber, amber-brown; III., amber.

α-Naphthol.—(A): I., purple, purple-black, brown-black; II., purple, dirty green-black, green-brown, purple-violet; III., sea-green, amber, pink-violet; IV., faintly pink. (A'): I., dirty green, brown, black-brown, brown-black; II., sea-green, brown-amber; III., yellow-green. (B): I., blue-green, indigo-blue, green-black; II., blue-green, dirty green. (B'): I., dirty green, deep brown; II., sea-green, dark amber-brown.

β-Naphthol.—(A): I., yellow-green, dark amber-brown, brown-black; II., green-amber, deep brown; III., amber; IV., straw-yellow. (A'): I., pink-brown, wine-red, deep red-brown; II., pink-amber, red-brown; III., pink-amber. (B): I., amber, yellow-green, green-brown; II., amber, brown-amber. *The naphthols are distinguished* best in (A) II. solutions; purple with the α, brown with the β.

Derivatives of Phenols.

Salicylic Acid.—(B), (B'): I., II., light chrome-green. Colour changed to amber by an excess of sodium hypobromite, restored to green by the addition of more ammonia.

Thymol, 2-isopropyl-5-methylphenol. (B'): I., II., brilliant nickel-green; III., faintly green colour. Excess of ammonia is to be avoided. This is a very beautiful and characteristic test for thymol.

Salol.—(B'): I., same as phenol and salicylic acid. Colour develops best after heating and standing.

Guaiacol, *o*-methoxyphenol. (A), (A'): I., deep red-brown; II., amber-brown. (B), (B'): I., deep blue-green or black-green; II., chrome-green. The colours of guaiacol resemble the colours of phenol more closely than of pyrocatechol.

Vanillin, 3-methoxy-4-oxybenzaldehyde. (B'): I., II., beautiful amber colour.

Arbutin.—(A): I., pink, amber; II., same. (B): I., deep nickel-green; II., same.

Eugenol, 4-propenyl-2-methoxyphenol.—(A), (A'), (B), (B'): I., II., golden yellow, changing to deep brown.

Oil of Bay, contains eugenol. (B'): I., clear deep red-brown; II., brown-yellow. Ammonia alone gives a bright lemon-yellow.

Oil of pimento, contains eugenol. (B'): I., red-amber, brown, deep red-brown; II., red-amber, brown, amber. Ammonia alone gives a bright lemon-yellow.

Scores of other fixed and volatile oils, tested as described above, failed to give characteristic colour tests. Homogeneous aromatic oils like safrol, apiol, and anethol failed to give the colour tests even after boiling for some time with concentrated alkalis. Under these conditions oil of winter-green gave the same green colour as salicylic acid.

Gallic Acid.—(A): I., pink, amber, brown, black-brown; II., pink, pink-amber, grass-green, amber-green, brown, amber; III., pink, brown, amber. Alkalis alone give first a pink, then a grass-green, and finally a brown colour, but the action is greatly quickened and intensified by means of sodium hypobromite.

Tannic Acid.—(A): I., II., pink, amber, brown, deep brown; III., pink, pink-amber, amber; IV., pink. Alkalis alone give first a pink, then a brown colour; here as with gallic acid, owing to the oxidising power of the hypobromite, the action is greatly quickened and intensified.

Aromatic Nitrogen Compounds.

Aniline.—(A): I., orange-red, deep red-brown; II., smoky amber, deep orange; III., amber, dirty orange; IV., light yellow. (A′): I., orange, deep brown; II., orange, deep brown; III., amber. With (A′) I. solutions, odour of phenylisocyanide.

o-Toluidine.—(A): I., brick-red, violet, purple, purple-black, deep brown-black; II., brown-red or orange-red; III., red-amber; IV., light straw-yellow. (A′): I., violet, purple, purple-black; II., yellow, pink-amber, brown-red; III., golden yellow. With (A′) I. solutions, odour of phenylisocyanide.

p-Toluidine.—(A): I., pink, rose-red, red-brown; II., pink, dirty yellow. (A′): I., violet, dark brown; II., pink-amber, golden brown.

These two toluidines and aniline are easily distinguished by the orange colour with aniline and by the changes of colour and deeper colours of *o*-toluidine.

p-Phenylenediamine.—(A): I., bright blue-green, deep purple, black-purple, brown-black; II., deep grass-green, deep purple, dirty brown; III., grass-green, amber-brown, violet-purple, curdy white precipitate; IV., bright amber. (A′): I., dark blood-red, purple, brown-black; II., deep brown-red; III., amber. (B′): I., II., deep purple, brown; III., brown amber. Very characteristic colour effects.

m-Nitraniline.—(A): I., yellow, dirty brown; II., ochre-yellow; III., light yellow. (A′): I., red-brown; II., dirty brown; III., light yellow.

p-Sulphanilic Acid.—(B′): I., deep red-brown; II., amber; III., light yellow.

Benzamide.—(A′): I., II., yellow, deep brown, brown-black, precipitate; III., amber-yellow. The colour is not easily discharged by an excess of hypobromite. An intense odour of phenylisocyanide is given off.

Phenylurethane.—(A′): I., brown or brown-red colour, odour of phenylisocyanide; II., cloudy yellow. (B′): I., orange, brown.

Phenylhydrazine.—(A′): I., II., dirty orange.

Ammonol, ammonia-phenylacetamide. (B′): I., yellow-red coloured precipitate; II., amber.

Fatty amides and amines fail to yield colour effects with sodium hypobromite, in fact no *aliphatic* compound responds to this reagent. It is specifically an *aromatic* test reagent.

Alkaloids.

Some forty of the most common alkaloids and glucosides were tested with sodium hypobromite; only the following ones gave definite colour reactions.

Morphine.—(A′), (B′): I., deep amber-brown; II., amber; III., light yellow. The test is best made with the use of ammonia; the colour is discharged by an excess of hypobromite.

Codeine and Dionine gave the same as morphine, only less sensitive.

Apomorphine.—(A): I., deep brown, deep chrome-green; II., pink-amber, deep brown, deep chrome-green, lighter green, amber; III., light pink, light yellow. This is a very characteristic reaction, particularly in solution II.

*Physostigmine or Eserine.**—(A): I., light red; II., pink. (A′): I., deep blood-red; II., bright bichromate-red; III., rose-pink. Alkalis give a red colour, but the hypobromite with heat deeply intensifies the red.

Antipyrine.—(A): I., pink-amber. Colour discharged by an excess of hypobromite.

Phenacetine.—(B′): I., deep amber-brown; II., amber.

Acetphenetidine.—(B): I., deep amber-brown; II., amber.

Though not giving characteristic colour effects, some of the other alkaloids gave off characteristic odours. For instance, scopolamine and hyoscyamine gave a phenylisocyanide-like odour, caffeine a methylamine odour, and atropine and cinchonidine other characteristic odours. From consideration of the above-described colour reactions and negative results obtained with scores of other organic groups, it is concluded that hypobromite is an invaluable reagent for detecting and distinguishing phenolic and aromatic amino compounds. None of the aliphatic or terpene compounds tested gave colour effects; only compounds of the types:—

$$>OH \qquad >NH_2 \qquad >CONH_2.$$

With *p*-phenylenediamine, it yields nearly quantitatively a white crystalline solid melting with decomposition at 105°. This and other products of the hypobromite reaction are being investigated.—*Journal of the American Chemical Society*, xxx., No. 9.

THE ELECTROLYTIC DETERMINATION OF BISMUTH.

By F. J. METZGER and H. T. BEANS.

Much has been written about the determination of bismuth in the electrolytic way and many electrolytes have been proposed. Without attempting any detailed discussion of the numerous publications which have appeared on the subject, the difficulties with which one almost invariably has to contend may be briefly summed up as follows:—

When a bismuth solution is electrolysed there is deposited not only metallic bismuth on the cathode, but frequently there is a simultaneous deposition of peroxide on the anode, and it has been suggested that both anode and cathode be weighed for each determination. Again, the deposited metal is nearly always black and spongy, and in cases where accurate results have been obtained it has been necessary to exercise the greatest care in washing and drying in order to prevent loss mechanically. A method which has been recommended especially for comparatively large quantities of bismuth is the amalgam method originally proposed by Vortman (*Ber.*, xxiv., 2749). In this method a known quantity of mercury salt is added to the bath and the deposit of bismuth amalgam weighed. From this weight the bismuth is obtained by difference. The quantity of mercury recommended is four times that of the bismuth present.

The method described in this paper presents none of the difficulties encountered in previous methods; the metal is deposited in compact, adherent form and can be washed and dried without any possible chance of loss, nor is there ever any deposition of peroxide on the anode when the electrolysis is completed.

It has been found that by the addition of acetic acid to a bismuth nitrate solution precipitation by hydrolysis may be completely prevented, even though the solution be subsequently largely diluted. It is also possible to obtain this result by first eliminating all free nitric acid by the addition of sodium hydroxide to alkaline reaction and then

* With bleaching powder, an intensely red colour; Ilber, *Zeit. Anal. Chem.*, xxviii., 134; Ferreira, *Bl.* (3), ix., 753.

Table I.

Expt. No.	30 per cent acetic acid. Cc.	Boric acid. Grms.	Volume. Cc.	N.D.$_{40}$ (*a*).	Voltage.	Temperature.	Time. Hrs.	Time. Mins.	Bi taken.	Bi found.	Error.
1.	20	2	250	0·2—0·15	2·1—2·8	74—79°	0	45	0·03956	0·0391	0·00046
2.	20	2	250	0·2—0·175	1·9—2·75	75—79	1	15	0·0989	0·0990	+0·0001
3.	20	2	250	0·2—0·15	1·9—2·8	72—75	1	30	0·0989	0·0990	+0·0001
4.	20	2	250	0·2—0·17	1·9—2·75	63—77	1	15	0·0989	0·0989	—
5.	20	None	250	0·2—0·15	1·9—2·7	74—78	1	15	0·0989	0·0989	—
6.	20	2	250	0·2—0·15	1·9—2·65	74—83	1	15	0·0989	0·0987	−0·0002
7.	20	2	250	0·2—0·15	1·85—2·74	72—77	1	15	0·0989	0·0989	—
8.	20	None	250	0·2—0·125	1·8—2·6	77—79	1	15	0·0989	0·0986	−0·0003
9.	20	2	250	0·2—0·2 (*b*)	1·9—2·8	75—78	1	15	0·0989	0·0984	−0·0005
10.	20	2	250	0·2—0·17	1·85—2·75	76—78	1	15	0·0989	0·0992	+0·0003
11.	20	2	250	0·2—0·17	1·8—2·72	76—79	1	15	0·0989	0·0992	+0·0003
12.	20	2	250	0·2—0·17	1·8—2·64	77—88	1	15	0·0989	0·0991	+0·0002
13.	20	None	250	0·2—0·175	1·8—2·4	75—78	1	15	0·0989	0·0990	+0·0001
14.	20	2	250	0·2—0·175	1·8—2·7	73—77	1	15	0·1978	0·1981	+0·0003
15.	20	2	250	0·2—0·16	1·8—2·6	76—77	1	15	0·1978	0·1979	+0·0001
16.	20	2	250	0·2—0·16	1·8—2·55	73—78	1	15	0·1978	0·1978	—
17.	20	2	250	0·2—0·17	1·77—2·55	70—80	1	15	0·1978	0·1980	+0·0002
18.	20	None	250	0·2—0·175	1·75—2·37	75—81	1	20	0·1978	0·1984	+0·0006
19.	30	None	250	0·2—0·16	1·8—2·45	70—78	2	15	0·3956	0·3962	+0·0006
20.	30	2	250	0·2—0·175	1·75—2·5	70—78	2	15	0·3956	0·3953	−0·0003
21.	30	None	250	0·2—0·15	1·7—2·42	73—79	3	15	0·3956	0·3955	−0·0001
22.	30	2	250	0·2—0·16	1·75—2·55	74—77	2	15	0·3956	0·3956	—
23.	30	2	250	0·2—0·175	1·72—2·45	74—79	2	15	0·3956	0·3957	+0·0001
24.	30	2	250	0·2—0·175	1·75—2·50	78—79	2	20	0·3969	0·3968	−0·0001
25.	30	None	250	0·2—0·175	1·76—2·55	75—80	3	30	0·3969	0·3969	—
				N.D.$_{100}$.							
26.	30	2	350	0·5—0·17	1·9—2·5	78—80	1	30	0·3969	0·3972	+0·0003

(*a*) The figures in the second column represent the point to which the current drops at the end of the experiment.
(*b*) In this experiment the current was maintained constant.

re-dissolving the precipitated bismuth hydroxide by means of acetic acid, the whole operation being carried out in the cold. It has also been observed that the addition of boric acid to the bath has a decided effect on the character of the deposit, and in addition it serves to bring about, readily, complete solution of bismuth hydroxide without the addition of very large quantities of acetic acid.

The apparatus employed is a slightly modified form of that originally proposed by Gooch and Medway (*Am. Journ. Sci.*, [4], xv., 320), using a rotating cathode. The platinum thimble employed as cathode was connected to the shaft by means of a rubber stopper wound with fine platinum wire for contact, and had an available surface of forty square centimetres. In all cases it was driven at the rate of about seven hundred revolutions per minute.

As a basis for the experiments a bismuth solution was prepared by dissolving chemically pure bismuth nitrate in water containing 25 cc. conc. nitric acid per litre. The solution was then carefully standardised by gravimetric methods.

The method of procedure was in all cases as follows:—To a known quantity of the standard solution phenolphthalein was added, then sodium hydroxide solution, drop by drop, to alkaline reaction. The precipitate formed was re-dissolved in acetic acid and then 2 grms. of boric acid were introduced. The solution was diluted, heated to 70° to 80°, and electrolysed. The working conditions are shown in Table I.

In Experiments 19 to 25 inclusive, the amount of acetic acid given does not completely re-dissolve the precipitate, but after the addition of the boric acid the solution becomes perfectly clear. In Experiments 19, 21, and 25, in which no boric acid was used, the slightly opalescent solution was electrolysed, the solution, however, becoming perfectly clear during the period of the electrolysis.

The change in voltage during the electrolysis serves as an excellent indicator in the determination. The voltage remains constant until all but the last trace of bismuth has been deposited, when it begins to rise rapidly to a maximum at which it again remains constant. The electrolysis should be continued five or ten minutes beyond this maximum point. In Table I. the voltages recorded show the initial and final values. Table II. being a record of Experiment 16 makes this a little more clear.

Table II.

Time, p.m.	N.D.$_{40}$.	Voltage.	Temperature.
2.50	0·2	1·8	73°
3.05	0·2	1.8	75°
3.20	0·2	1·8	76°
3.35	0·2	1·8	78°
3.43	0·19	2·0	78°
3.45	0·18	2·1	78°
3.50	0·175	2·4	78°
3.55	0·175	2·5	78°
4.00	0·160	2·55	78°
4.05	0·160	2·55	78°

The action of acetic acid on the deposited metal was found to be so slow that it is not necessary to syphon off the liquid at the end of the experiment. All that is required is to quickly substitute a beaker of water without interrupting either the current or rotator. The thimble is then detached, washed with alcohol and ether, allowed to dry, and weighed after standing on the balance for ten minutes. To avoid any errors which might arise from changes in atmospheric conditions, the weight of the thimble was taken after dissolving off the deposited metal by immersing in nitric acid, washing and drying, &c., the same as above.

In all of the experiments given in Table I. the available cathode surface was forty square centimetres, and it was naturally supposed that a stronger current could be employed with a larger electrode, and in this way the time factor might be reduced. A larger electrode having an available surface of one hundred square centimetres of the same form as the one previously used was procured with which we obtained the result shown in Experiment 26.

In working with this larger electrode the current should be reduced to 0·2 ampère at the time whe begins to rise.

As seen in the table the results are accurate without the use of boric acid. The metal is deposited in bright, compact form up to the time when the voltage begins to rise, *i.e.*, when only very small quantities remain in solution. From this point, if there be no boric acid present, the last traces of metal deposit over the surface of the bright coating in the form of a granular layer less firmly adherent and somewhat darker. With boric acid present the deposition is uniform to the very end of the experiment, giving a mooth, compact, adherent deposit having the characteristic colour of the metal.

In not a single experiment was there any evidence of peroxide formation on the anode at the close of the determination. Occasionally there was a slight tinge of yellow on the anode at the very beginning of the electrolysis, but this, however, soon disappeared. The solution after each determination was examined for bismuth and none could be found.

The effect of boric acid on the deposition of other metals is being studied.—*Journal of the American Chemical Society*, xxx., No. 4.

FIFTEENTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED DURING 1907.*

By F. W. CLARKE.

(Concluded from p. 193).

Tellurium.

The research upon tellurium, by Baker and Bennett (*Journ. Chem. Soc.*, xci., 1849), was primarily to determine the homogeneity of the element. Various fractionating processes were employed, but tellurium of the same atomic weight was produced in every case. Two methods, both new, were adopted for the atomic weight determinations. First, TeO_2 was heated with sulphur in tubes of glass, the two ends of the tube being packed with pure silver leaf. Sulphur dioxide was expelled, and from its amount, as measured by the loss of weight, the percentage of oxygen in the TeO_2 was computed. For this purpose the value S = 32·06 was assumed. The determinations by this method fall into three principal series, as follows, representing differences in the source of the initial substance:—1. Fractional crystallisation of telluric acid from barium tellurate. 2. Fractional crystallisation of telluric acid from oxidation of the element. 3. Tellurium dioxide prepared from tellurium hydride. Vacuum weights are given throughout. The data are as follows:—

Fraction.	Weight TeO_2.	Loss SO_2.	Per cent O in TeO_2.
	SERIES 1.		
1.	1·51509	0·60838	20·055
2.	1·09875	0·44074	20·034
3.	1·02150	0·40993	20·046
4.	0·90835	0·36472	20·053
5.	1·00702	0·40451	20·062
6.	1·01515	0·40733	20·044
	SERIES 2.		
1.	1·56837	0·62938	20·046
2.	1·07852	0·43257	20·035
3.	1·72627	0·69296	20·052
4.	2·09253	0·83927	20·032
5.	0·83335	0·33465	20·059
6.	1·15372	0·46284	20·041
7.	1·68618	0·67661	20·045
8.	0·90835	0·36472	20·053
	SERIES 3.		
1.	1·02217	0·41050	20·064
2.	0·80697	0·32322	20·051
3.	1·32003	0·52992	20·053
4.	1·05207	0·42221	20·047
5.	1·37043	0·54969	20·032
6.	0·95944	0·38511	20·048

* From the *Journal of the American Chemical Society*, xxx., No. 3.

Several other experiments, concordant with these, are cited, but without the detailed weighings.

The other method employed consisted in converting tellurium into the tetrabromide by direct union with bromine in an atmosphere of nitrogen. Here, again, several series of determinations are given. For brevity, the data are combined in one table:—

Series.	Weight Te.	Weight $TeBr_4$.	Per cent Te.
1.	0·61273	2·14933	28·508
	0·56866	1·99354	28·525
	0·59884	2·09951	28·523
	0·57894	2·03040	28·514
	0·54743	1·91899	28·527
	0·33859	1·18732	28·517
	0·56866	1·99354	28·526
	6·47643	1·67025	28·525
	0·56622	1·98597	28·511
2.	0·44271	1·55205	28·524
	0·41671	1·46177	28·508
	0·50611	1·77489	28·515
3.	0·37382	1·31081	28·519
	0·31895	1·11868	28·512
	0·48931	1·71554	28·522
	0·47156	1·65404	28·510
4.	0·40748	1·42867	28·523
	0·62013	2·17449	28·518
5.	0·37382	1·31081	28·519
	0·50822	1·78207	28·518
	0·12928	0·45354	28·505
	0·42926	1·50540	28·515
6.	0·80348	2·81715	28·511
	0·95309	3·34193	28·512

From the dioxide determinations, the authors compute that Te = 127·609. From the bromide syntheses, if Br = 79·96, Te = 127·601. They also give, but without details, several determinations based upon analyses of tellurium tetrachloride. Four fractions of the chloride gave for Te the values 127·58, 127·60, 127·64, 127·62. The commonly accepted value, 127·6, thus receives strong confirmation.

On the other hand, quite different results have been announced by Marckwald (*Ber.*, xl., 4730). He prepared pure telluric acid, H_6TeO_6, which, by heating under proper precautions, was reduced to TeO_2. His figures, on the basis of O = 16 and H = 1·008, are subjoined.

Weight acid.	Weight TeO_2.	At. wt. Te.
8·6277	5·9884	126·93
12·2680	8·5135	126·84
13·0051	9·0244	126·80
8·6415	5·9947	126·65
8·4588	5·8696	126·80
8·0113	5·5599	126·94
	Mean, excluding No. 4 ..	126·86

From the sums of the weights Marckwald, rejecting the fourth determination, computes Te = 126·85. This falls below the atomic weight of iodine, and is in harmony with the periodic law. Since, however, it diverges so widely from many, concordant, higher determinations, it evidently needs corroboration by other experimenters and other methods.

Ytterbium and Lutecium.

According to Urbain, ytterbium is a mixture of two elements, neo-ytterbium and lutecium (*Comptes Rendus*, cxlv., 759). The atomic weight of neo-ytterbium is not far from 170; that of lutecium is perhaps a little above 174. Detailed determinations are yet to be made. In a footnote Urbain remarks that the atomic weight of thulium, which is given as 171 in the tables, is certainly below 168·5.

Radium.

Madame Curie has re-determined the atomic weight of radium, with purer material than that used in her former researches, and in larger quantities (*Comptes Rendus*, cxlv., 422). The well known chloride method was employed, with the subjoined results :—

Weight $RaCl_2$.	Weight Ag Cl (*a*).	At. wt. Ra.
0·4052	0·3906	226·35
0·4020	0·3879	226·04
0·39335	0·3795	226·15
	Mean	226·18

(*a*) From the weights of AgCl, 0·00006 grm. is to be deducted in each case, representing filter ash.

Calculated with Ag = 107·8 and Cl = 35·4. With Ag = 107·93 and Cl = 35·45, Ra becomes 226·45, or 226·5 in round numbers. It is announced that Thorpe is also engaged upon a re-determination of this constant.

Gaseous Densities.

Gray (*Proc. Chem. Soc.*, xxiii., 119) has determined the density of gaseous hydrochloric acid, and assigns to the weight of 1 litre, under standard conditions and in latitude 45°, the value 1·6397 grms.

Guye (*Comptes Rendus*, cxlv., 1164) has discussed the data relative to nitrogen and some of its gaseous compounds, and assigns to it the atomic weight 14·010. D. Berthelot (*Comptes Rendus*, cxlv., 65), from essentially the same data finds N = 14·005. In an elaborate discussion of the whole subject, Guye has finally adopted the following figures for the weight in grms. of 1 litre of each gas considered. (See *Arch. Sci. Phys. Nat.*, [4], xxiv., 34; *Journ. Chim. Phys.*, v., 203; *Comptes Rendus*, cxliv., 976; Chemical News, xcvi., 175; *Journ. Am. Chem. Soc.*, xxx., 143. Other papers, mostly controversial, are as follows: Guye, *Comptes Rendus*, cxliv., 1360; Berthelot, *Ibid.*, cxliv., 76, 269) :—

O_2	1·42900	N_2O ..	1·9777
H_2	0·08987	HCl . ..	1·6398
N_2	1·2507	NH_3 ..	0·7708
CO	1·1504	SO_2 . ..	2·9266
NO . ..	1·3402	Air	1·2928
CO_2 . ..	1·9768		

Miscellaneous Notes.

Watson (*Nature*, lxxvii., 7), in a brief note, has considered the simultaneous calculation of atomic weights from a group of related ratios. Hinrichs (*Comptes Rendus*, cxlv., 715) has proposed a graphic method for achieving the same purpose. He has also published several papers in which he seeks to establish integral values for the atomic weights of Br, Mn, and Cl (*Comptes Rendus*, cxliv., 973, 1343; cxlv., 58; see also *Chem. Zentralb.*, 1907, p. 1958).

A number of writers have discussed relations between the atomic weights. (See Wilde, *Manchester Lit. Phil. Soc.*, li., [1], No. 2; Stromeyer, *Ibid.*, No. 6; Minet, *Comptes Rendus*, cxliv., 428; Collins, Chemical News, xcvi., 176; Verschoyle, *Ibid.*, xcvi., 197; Delauny, *Comptes Rendus*, cxlv., 1279). On the question of standards, see Erdmann (*Chem. Zeit.*, xxxi., 95), and especially Swarts (*Bull. Acad. Roy. Belg.*, Classe des Sciences, 1907, No. 3, p. 212). The latter proposes two methods by which the atomic weights may be connected with the gas equation and with the absolute system of units. General papers on the subject of atomic weights are by Brauner (*Chem. Zeit.*, xxxi., 483) and Richards (*Ber.*, xl., 2767). Richards's paper is in the form of a lecture delivered before the German Chemical Society.

Lectures on Milk.—Dr. Harden, of the Lister Institute of Preventive Medicine, will give a course of Six Lectures on "Milk," at the South-Western Polytechnic, on Thursday evenings, commencing October 29, at 8 p.m. Each lecture will be illustrated by specimens and lantern slides and will appeal to managers and others interested in the distribution of milk.

THE INFLUENCE OF FINE GRINDING ON THE WATER AND FERROUS-IRON CONTENT OF MINERALS AND ROCKS.*

By W. F. HILLEBRAND.

It has been the general practice, following the directions of numerous writers, to reduce most rock and mineral powders to a very fine state of division preliminary to their analysis. This seemed called for on several grounds—to secure a proper sample of an inhomogeneous mixture, the need of extreme fineness for the ferrous iron determination, for that of the alkalis by the J. Lawrence Smith method, for all decompositions that involve the use of boric oxide or lead oxide, and in general in order to insure complete and speedy decomposition by fusion with an alkali carbonate. A very fine powder is, however, far less needed for the last-named purpose than has been commonly supposed. The writer's recommendation has been to prepare a main sample of moderate fineness and to grind separate small portions of this finer when needed.

Now comes the Swedish chemist, Robert Mauzelius, and shows that fine grinding is highly detrimental in the case of minerals containing ferrous iron because of its resulting oxidation (*Sveriges Geol. Undersökning Arsbok*, 1907, i., No. 3). Led by Mauzelius's work, Nicholas Knight has shown that siderite oxidises when finely ground (Chemical News, xcvii., 122). A. L. Day and E. T. Allen had shown how very great is the effect of fine grinding on the moisture content of minerals (*Pub. Carnegie Inst.*, No. 31, p. 56; *Am. Journ. Sci.*, xix., 93; *Bull. U. S. Geol. Survey*, cccv., 55); and Mauzelius in the paper cited adds to our knowledge on this point by quantitative data with respect to rock powders. These writers clearly show that coarse powders entirely free from hygroscopic water, or containing but a few hundredths of 1 per cent of it, take up moisture progressively as the grinding continues, and that the amount so taken up can readily be brought to exceed 1 per cent of the weight of the sample. This is, so far as amorphous minerals are concerned, entirely in harmony with what Bunsen observed for spun glass (*Wied. Ann.*, 1885, xxiv., 327), a condition in which the surface exposure is very great. Since many minerals and nearly all rocks contain ferrous iron, Mauzelius rightly insists that the determination of this component shall be made on the coarsest material that can be completely decomposed by the acid that is employed. Further, in view of the great absorption of atmospheric moisture, and in such varying degree according to the fineness of the sample that no two analysts can be expected to prepare samples of the same mineral that will contain like amounts of moisture, he argues that the old practice of stating the analysis in terms of dried material should be followed in preference to the later practice of reporting the moisture given off below 100—110° as well as that firmly held at that point. To this last recommendation objection may be made, as I shall show later.

The oxidation of ferrous iron was such an important observation in view of the extent to which it may go (up to about 40 per cent of the FeO present in one of Mauzelius's experiments) that the subject seemed to demand further investigation. It was evident that acceptance of the direction to use only relatively coarse powders would introduce serious complications and uncertainties into the execution of an analysis, for there are many minerals that cannot be readily decomposed by boiling hydrofluoric acid even when reduced to pass a sieve of 150 meshes to the linear inch (nearly 60 to the centimetre, or approximately 0·08 mm. width of apertures between wires). It was therefore necessary to devise, if possible, some means of fine grinding that would not involve oxidation of iron.

Much of the tabulated matter of this paper is but a confirmation and extension of what Allen, Day, and Mauzelius have shown, but its presentation in detail seems justified

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* From the *Journal of the American Chemical Society*, xxx., No. 7.

on account of the intimate bearing of the subject on mineral analysis and its consequent importance to all mineral analysts, mineralogists, and petrologists. Often valuable observations have passed unheeded or insufficiently heeded, and been eventually forgotten. Frequent reiteration is called for if they are to become generally known and appreciated.

In the course of the experiments that were made on powders ground in air, no explanation that occurred seemed adequate to fully account for the oxidation of ferrous iron except that of strong local heating of the grains at the moment of fracture or attrition under the pestle. The effect should be especially marked with pyritic minerals. The idea of intense local heating, though at first thought improbable, becomes less so when it is remembered that steel strikes sparks from flint. If the oxidation is due simply to greater surface exposure it ought seemingly to continue at a lessened rate after cessation of the grinding, but powders finely ground in past years show to-day the same FeO content as when originally analysed. Of course, there is no general heating to account for the phenomenon, as when a rock is pulverised in a closed ball mill, where the temperature may rise to such a degree as to dehydrate the gypsum that is added to Portland cement clinker (see Note 1).

Led by the thought of local heating as the chief cause of oxidation of the ferrous iron, it occurred to me to test the effect of grinding on the content of water that is given off above 100—110°, and is supposed to be chemically held in large part, for it seemed possible that this might be reduced by grinding. If so, however, the loss is more than offset by a very large gain due to increased surface exposure, as the results tabulated below plainly show. This gain in firmly held water, quite in accord again with what might be expected in view of the firmness with which Bunsen found (*loc. cit.*) the adsorbed water to be held by glass, has an important bearing on the question of the permissible size of grain of a sample that is to be analysed, and strengthens in the strongest manner the argument of Mauzelius for a coarse sample. For the rock powders tested by myself, the increase in this firmly held water seems to be, as a rule, of nearly the same general order of magnitude as that of the loosely held moisture, but it is very much greater with the meerschaum tested (16 below).

The following remarks explain certain features of the table. The determinations of the first three numbers were made simply to test the claims of Mauzelius, those that follow relate also to increase of water above 105° under different conditions of grinding. The designations <100-mesh, <150-mesh (see Note 2), &c., mean that all the material was simply crushed to pass the respective sieves with as little production of fine powder as possible and without grinding. This was effected by brief crushing in the steel mortar figured on p. 46 of *Bulletin*, cccv., *of the U. S. Geological Survey*, then sifting, transferring the coarser matter again to the mortar, and repeating these operations till all had passed the sieve. Nevertheless, there was unavoidably much powder formed that passed 150 meshes and even 200 meshes to the inch. Material thus prepared and analysed was then ground in air, and also under water, absolute alcohol, or carbon tetrachloride, sometimes all three, in order to test the effect of these liquids, both on oxidation of iron aud absorption of water. The twenty-minute and thirty-minute grindings were all done by hand, those of longer duration by the McKenna mechanical grinder. Grinding by hand is less effective in producing fine powder than by a mechanical grinder for the same length of time, and different grinders may be very unequally effective (6, *c*, *d*). In comparing figures in the table it should be remembered that equal periods of grinding do not necessarily result in the production of powders of the same fineness, especially as between dry and wet grinding. Where more than one determination of water or iron was made on a given sample all are reported in order to show, more particularly, the generally excellent agreement of the iron values on a given sample when the rocks are readily decomposable and the variations to be expected when they are not. In the latter cases, finer grinding would have led to closer agreement but lower values.

All water percentages not otherwise indicated are based on direct weighings. If it were important to show the actual amount of water taken up by a given weight of unground material, the mode of statement would have been otherwise than by percentages of those weights. To give the percentages of ferrous oxide actually found would convey an incorrect impression. They have therefore been corrected by raising them in proportion to the amount of water absorbed during grinding, so as to be properly comparable with the percentages in the unground material, and give a true picture of the extent of oxidation suffered by the latter. These corrections range from 0 to 0·13 per cent. The same result would have been reached by reducing all FeO percentages to an anhydrous basis. In 1, 2, and 3 the FeO percentages in ground material are still too low, the full correction not being applicable for lack of data as to how much absorbed water was held at 110°.

Notes.

1. R. K. Meade, "Portland Cement," p. 307. In connection with the dehydration of gypsum, an interesting experiment made by Dr. E. C. Sullivan and Mr. Geo. Steiger in the laboratory of the U.S. Geological Survey, Washington, D.C., may well receive mention. They were able by grinding several hours daily for several days with a mechanical grinder to reduce the water of crystallisation of gypsum from 20 per cent to about 5 per cent, without evidence that a limit had been reached. It is regrettable that no effort was made to ascertain if this lost water could be regained by exposure to the air after grinding.

2. The following are the approximate diameters of the apertures in the sieves used:—

Mesh.	Mm.
80	= 0·20
100	= 0·18
120	= 0·11
150	= 0·08
200	= 0·065

These sieves were of metal, the use of which, in preparing samples for full analysis, is forbidden if traces of metals like those of which the sieve is made are to be looked for. In such cases, sieves of the best silk bolting-cloth shaken in a very gentle manner should be used.

(To be continued).

THE COMPARATIVE OXIDISING POWER OF SODIUM PEROXIDE AND ITS USE IN QUALITATIVE ANALYSIS.

By D. F. CALHANE.

The detection of chromium in qualitative analysis rests on its oxidation to the chromate and the precipitation of chromate of lead in acetic acid solution. The oxidation is usually effected by chlorate of potash on the solution of chromium hydroxide in strong nitric acid. If by any chance the nitric acid chosen is not strong enough or has become diluted, the oxidation will not occur, and the test fails. It is necessary for success that the hydroxides possibly containing iron, aluminium, and chromium be freed of water as much as it is possible by drying them quickly by heating. In this part of the procedure the student usually fails to work properly, not enough water is removed, and the subsequent oxidation does not occur. As I have found, in teaching classes in this subject, so much trouble in getting the student to properly observe precautions, it seemed of advantage to apply a test which would be effective and not so much dependent on a certain set of conditions.

Some experiments were accordingly made on the action,

of certain other oxidising agents. Among these were chosen sodium peroxide and bromine water. The preference was for the former, as it is to be obtained in a convenient solid form, and possesses few of the disagreeable features of bromine water. A 0·1 N solution of chrome-alum was prepared, each cc. holding 0·0332 grm. of chrome-alum, answering to 0·005 grm. of Cr_2O_3. Next, the oxidation of portions of this solution with peroxide of sodium and bromine water was carried out to see what the comparative efficacy of these two reagents is on solutions containing known amounts of chromium. In this connection an interesting feature in the action of sodium peroxide on chromium solutions was met with, that at first led to the belief that bromine water was more efficient. The dilutions were carried to the point where the amount of chromic oxide present was only 0·000125 grm. corresponding to 0·000085 grm. of chromium. At this extreme dilution, a safe test was secured with bromine water, but the sodium peroxide apparently failed at a concentration answering to 0·00025 grm. chromic oxide. The bromine water appeared to act equally well both hot and cold at all the different concentrations from 0·005 grm. chromic oxide down to 0·000125 grm. With the sodium peroxide there was no test with the presence of 0·005 grm. chromic oxide, if the solutions were treated cold. When the sodium peroxide was added to the hot solution a small amount of yellow precipitate was obtained. An explanation of this surprising fact was arrived at later.

The next procedure was to find out what the limit of delicacy for the test is in actual analysis. Normal solutions of aluminium nitrate and ferric chloride were prepared, 1 cc. of which answered to 0·071 grm. aluminium nitrate and 0·054 grm. ferric chloride, giving a precipitate of 0·026 grm. aluminium hydroxide, 0·0356 grm. ferric hydroxide, and 0·0033 grm. chromic hydroxide from 1 cc. of the chrome-alum. The analysis in the first instance was carried through with a mixture of 1 cc. of each of the three solutions. The aluminium and iron here exceeded the chromium in the ratios of 8 and 12 times, respectively. The solution of the mixed salts was treated with 10 cc. of barium carbonate emulsion, and allowed to stand for two or three minutes. Next a filtration was made, and the filtrate tested for iron, chromium, and aluminium. The tests proved complete precipitation of the metals. The barium carbonate residue was dissolved in hot hydrochloric acid. The hydroxides of the three metals re-precipitated with ammonia. These were dissolved in hydrochloric acid, and the diluted solution divided into two equal parts. There could be present in each part the equivalent of 0·0025 grm. chromic oxide, answering to 0·0016 grm. chromium hydroxide. One part was added to a large excess of bromine water, after previously having added excess of sodium hydroxide. After filtration from the iron, no test for chromium appeared on adding acetic acid and lead acetate. In the other portion similarly treated, using sodium peroxide as the oxidising agent, a good test was obtained. Next, the above procedure was repeated, using 2 cc. of the iron and aluminium solutions and 1 cc. of the chromium. The ratios here were 24 iron, 16 aluminium, to 1 of chromium hydroxide. Here, again, the bromine water failed to act sufficiently for a test, while the sodium peroxide gave a good test. In the third experiment 5 cc. each of the iron and aluminium solutions were taken, and 0·5 cc. of the chrome-alum solution. After proceeding as before, the bromine water failed to give a test, while 0·5 grm. sodium peroxide gave a reliable indication. The ratios here were 81 aluminium and 111 iron to 1 of chromium. The mixed precipitate contained 0·130 grm. aluminium hydroxide, 0·178 grm. ferric hydroxide, and 0·0016 grm. chromium hydroxide. In each of the last two tests the maximum amount of chromic oxide present for oxidation was 0·0008 grm., corresponding to 0·0005 grm. chromium.

In the next experiment it was desired to contrast the delicacy of the usual method with potassium chlorate and nitric acid with the peroxide procedure. Amounts taken were 5 cc. aluminium nitrate, 5 cc. ferric chloride, and 0·5 cc. chrome-alum. The analysis was carried through in the usual way, and the precipitate of iron, chromium, and aluminium hydroxides given by ammonia was dissolved in strong nitric acid, and boiled with potassium chlorate. The solution was divided into two equal portions, and one tested with sodium hydroxide acetic acid and acetate of lead. The result was negative for chromium. Here, as in the previous cases, 0·0008 grm. of chromium hydroxide was present. The result shows that the limit of successful oxidation by this method has been exceeded. The time required to carry this out is longer and the test is much less delicate than with the peroxide method.

Another test was made to compare the fusion method of oxidation with that using bromine water, potassium chlorate, and nitric acid and sodium peroxide. The usual method of fusing the hydroxides of iron, chromium, and aluminium mixed with dry potassium nitrate and sodium carbonate on platinum was followed, having present the same amount of chromium, iron, and aluminium as in the previous cases. The result here for chromium by the usual test as lead chromate after oxidation was surprisingly convincing. Five cc. of the ferric chloride aluminium nitrate and 0·5 cc. of the 0·1 N chrome-alum solution were precipitated by ammonia. The precipitate was filtered, washed, and fused in a platinum crucible with potassium nitrate and sodium carbonate mixture for a few minutes. The cooled mass was lixiviated with hot water. The iron was filtered off, and one-half of the clear filtrate, which was decidedly yellow, was made acid with acetic acid and lead acetate added. A good yellow colour was produced due to the formation of lead chromate. This test appears to be equally delicate with the sodium peroxide method. There was present in this fusion test 0·0008 grm. chromic oxide, or 0·0005 grm. chromium.

In the experiments having to do with the oxidation of the pure chrome-alum solution with bromine water and peroxide, it was surprising that the peroxide apparently failed to oxidise the chromium at a concentration where bromine water gave excellent results. It was, found, however, that if the solution was hot during the test a very slight precipitate of lead chromate was obtained with the sodium peroxide procedure. Bromine water acted equally well in hot or cold solution. It has been previously shown that in actual analysis sodium peroxide is by far the more powerful oxidising agent.

It was found on further experimentation that whereas a solution of bichromate was readily oxidised to perchromate by barium peroxide in acid solution, a chromic salt gave no indication of oxidation to the perchromate by the above agent. The chromic salt was only slightly oxidised to the chromate.

Sodium peroxide acting on a solution of the chromic salt gave a yellow colour. On acidification a violet colour appeared, which concentrated in ether indicated perchromic acid. In acid solution sodium peroxide gave no such oxidation. If a chromic salt were oxidised by sodium peroxide as above, the solution acidified with acetic acid, and lead acetate added, in dilute solutions such as were used in this work, no precipitate appeared. On allowing the solution to stand, or more quickly, on warming, the customary yellow precipitate of lead chromate appeared, and oxygen continued to be evolved for some time.

These results show the reason of the failure to obtain the tests for chromate of lead in the earlier part of the work where dilute solutions of chrome-alum were severally treated with sodium peroxide and bromine water. The sodium peroxide forms an alkaline solution with the evolution of oxygen. The chromic salt is oxidised to the perchromate, the sodium salt being formed in the alkaline solution. This substance is stable, and gives a yellow colour to the solution. On acidification with acetic acid, lead acetate produces no precipitate, as the lead perchromate is soluble in this medium. On standing or warming, oxygen is given off, and the lead perchromate breaks down to the chromate, giving the customary yellow precipitate.

Oxone, the fused form of sodium peroxide, acts the same

as the unfused variety, as would be expected. For analytical work it is less desirable, owing to the impurities it contains. In addition to the silica found in the unfused variety, it carries about 1·5 per cent of copper, as the result of several analyses showed. In addition small amounts of iron were found to be present. The results obtained in this investigation show that sodium peroxide is the best oxidising agent for chromium in solution. Sodium perchromate is formed by this agent in alkaline solution. This solution is stable, and, on acidification, oxygen is rapidly and continuously evolved. The oxygen can be liberated as fast as desired, thus giving a powerful oxidising source simply controlled.

The properties of this alkaline perchromate solution will be further investigated.—*Journal of the American Chemical Society*, xxx., No. 5.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 196).

Confirmatory Experiments and References (continued).

G. D., Section 6: Precipitation of Vanadium by Alkaline Hydroxides when Uranium is present.—For the insolubility of uranyl vanadate, see v. Klecki (*Zeit. Anorg. Chem.*, 1894, v., 381), Carnot (*Comptes Rendus*, 1887, civ., 1850); also C. E., *P. 58*a*, N. 3.

Fifty mgrms. V as Na_3VO_4 and about 250 mgrms. U as $UO_2(NO_3)_2$ were dissolved in 3 or 4 cc. HNO_3 (1·20) and 30 cc. water; the solution was made alkaline with NH_4OH (0·96); a large yellow precipitate separated. This was filtered off, and the solution was tested for vanadium by *P. 58*e*; not more than 1 mgrm. was found. In this experiment there was just sufficient uranium to form $UO_2NH_4VO_4$. The precipitate obtained in the preceding experiment was dissolved in a little HNO_3, diluted to about 15 cc., and poured into about 20 cc. 10 per cent NaOH solution; a large yellow precipitate separated. This was filtered off, and the filtrate tested for vanadium by *P. 58*e*; only 5 to 10 mgrms. V were found.

Separation of Zinc and Vanadium by Hydrogen Sulphide.—H_2S was passed into a solution containing H_3VO_4 and acetic acid; the solution slowly became blue, and sulphur separated, which rapidly became dark coloured owing to the precipitation of some V_2S_4.

One mgrm. V as Na_3VO_4 and 50 mgrms. Zn as $Zn(NO_3)_2$ were dissolved in 30 cc., a little acetic acid was added, and an excess of about 3 cc. NH_4OH (0·90). The clear solution was saturated with H_2S. A heavy precipitate of ZnS separated at once, and the solution slowly became deep red in colour, which colour was very distinct after filtering. The experiment was repeated with 1 mgrm. Zn and 50 mgrms. V; a precipitate of ZnS was obtained, which was confirmed by the last paragraph of P. 57.

Action of NH_4OH *on Uranium Solutions containing* H_2O_2.—100 mgrms. U as $UO_2(NO_3)_2$ were treated by P. 52. The red (peruranate) solution was made acid with HNO_3. Without heating, the acid solution (which contained H_2O_2, see C. E., P. 52, N. 6) was made alkaline with NH_4OH; a large yellow precipitate of $(NH_4)_2U_2O_7$ separated, but the filtrate was yellow. The filtrate was acidified with HNO_3, boiled for two or three minutes, and again made alkaline with NH_4OH; a large yellow precipitate separated, which was nearly as large as the first NH_4OH precipitate. The colourless filtrate was again acidified, boiled, and then made alkaline with NH_4OH; no precipitate separated. Therefore uranium, if present, will divide in P. 53.

To a nitric acid solution containing 200 mgrms. U as $UO_2(NO_3)_2$ and 40 mgrms. V as H_3VO_4 in 40 cc. were added about 20 cc. 3 per cent H_2O_2; the mixture was made alkaline with NH_4OH; no precipitate separated, showing that the precipitation of $UO_2NH_4VO_4$ and of $(NH_4)_2U_2O_7$ is prevented by the presence of an excess of H_2O_2.

Action of $BaCl_2$ *on Uranate Solutions.*—100 mgrms. U as $UO_2(NO_3)_2$ were treated by P. 52 and 53. The yellow filtrate from the NH_4OH precipitate, which contained about half the uranium, was treated by P. 55; a moderately large precipitate separated. After standing over-night the precipitate was filtered off, and the filtrate tested for uranium by adding HNO_3, boiling, and adding NH_4OH; a small yellow precipitate separated, which was estimated to contain 2 mgrms. U. Therefore barium uranate (or peruranate) may be precipitated nearly completely in P. 55.

Action of $BaCl_2$ *on Vanadate Solutions.*—100 mgrms. V as Na_3VO_4 were treated by P. 52 and 53. See C. E., P. 52, N. 10. The NH_4OH solution, which had a volume of about 40 cc., was made just acid with acetic acid, and $BaCl_2$ solution was added in the cold as described in P. 55; no precipitate separated from the yellow solution in five minutes. The experiment was repeated with 300 mgrms. V; no precipitate appeared when the $BaCl_2$ was first added, but in ten minutes a large orange precipitate of barium vanadate had separated. This was filtered off, and the filtrate allowed to stand over-night; a large precipitate formed. The precipitate was collected on a filter and washed with water; much of it dissolved, showing that barium vanadate is fairly soluble in water. A portion of the filtrate which was still slightly yellow was tested for vanadium by *P. 58*e*; much vanadium was found, showing that the precipitation of barium vanadates take place slowly, and that large amounts of vanadium remain in the filtrate in the cold. A solution containing 100 mgrms. V as Na_3VO_4 and a little NH_4OH in 40 cc. was made acid with acetic acid; 10 cc. 10 per cent $BaCl_2$ solution were added, and the mixture was boiled for a minute or two; a large nearly white precipitate separated, and the solution became perfectly colourless. The precipitate was filtered off, and the filtrate tested for vanadium by *P. 58*e*; the filtrate was found to contain not more than a few mgrms. of vanadium, thus showing that a large proportion of the vanadium had been precipitated on boiling. The last experiment was repeated, except that the acetic acid solution was made alkaline with NH_4OH, before boiling; the filtrate was found to be nearly free from vanadium. The last experiment was repeated, with a mixture of 100 mgrms. V and 10 mgrms. Zn as nitrate; only about 3 mgrms. Zn were found in the filtrate, showing that a large part of it had been precipitated with the vanadium, probably as zinc vanadate.

Action of NH_4OH *on Solutions containing Zinc and Vanadium as Vanadate.*—500 mgrms. Zn as nitrate and 100 mgrms. V as Na_3VO_4 were treated by P. 52; only a very small precipitate remained, and the solution was colourless. The filtrate was acidified with HNO_3 (1·20), an excess of about 2 cc. being added, and the solution was made just alkaline with NH_4OH; a large precipitate separated. Four cc. NH_4OH (0·96) were added; the precipitate dissolved completely. The NH_4OH solution was neutralised with acetic acid; a large precipitate separated, but it dissolved when a small excess of acetic acid was added. The acetic acid solution was boiled for a minute or two; a large precipitate separated, leaving a clear solution, which, however, still contained considerable zinc and vanadium.

Separation of Zinc and Uranium by NH_4OH.—One mgrm. Zn as $Zn(NO_3)_2$ and 100 mgrms. U as $UO_2(NO_3)_2$ were dissolved in 4 cc. HNO_3 (1·20) and 100 cc. water; the mixture was made alkaline with NH_4OH and an excess of about 3 cc. added; the yellow precipitate was filtered off, and the solution tested for zinc with H_2S; a small

* From the *Journal of the American Chemical Society*, xxx., No. 4.

nearly white precipitate separated. The experiment was repeated with 50 mgrms. Zn and 100 mgrms. U; the filtrate contained nearly all the zinc. The precipitate was washed, dissolved in HNO_3, diluted to 100 cc., and treated again with NH_4OH; the filtrate contained only about 2 mgrms. Zn. The NH_4OH precipitate was again treated in the same way; the filtrate contained no zinc.

Action of NH_4OH *on Solutions containing Uranium and Chromate.*—Four mgrms. Cr as K_2CrO_4 and 100 mgrms. U as $UO_2(NO_3)_2$ were treated as described in the preceding paragraph; the filtrate from the NH_4OH precipitate contained nearly all the chromium; the second NH_4OH filtrate was colourless. The experiment was repeated with 50 mgrms. Cr and 100 mgrms. U; the filtrate after the second NH_4OH precipitation was nearly colourless, thus showing that little or no chromium was carried down with the uranium.

G. D., Section 13: *Separation of Titanium and Zirconium by Boiling with Sulphurous Acid.*—With regard to the unsatisfactory nature of the process usually employed, see Hillebrand (*Bull. U. S. Geol. Sur.*, 1900, clxxvi., 72). A fairly satisfactory quantitative separation of titanium from iron is obtained by boiling a solution of a volume of about 1 litre containing acetic and sulphuric acids. This large volume is impracticable in qualitative analysis, and H. I. Wood and B. E. Schlesinger (Theses, Massachusetts Institute of Technology, 1901, 1902) devised the following method:—"Heat the HCl solution (volume 10 to 20 cc.) to boiling, remove the flame, add at once carefully NH_4OH until litmus-paper is turned distinctly blue, add 30 to 40 cc. H_2SO_3 solution, and place on the water-bath for fifteen to twenty minutes." This method was carefully tested by us. The results were satisfactory with titanium or zirconium alone, and small quantities of these elements were easily detected. But when iron was also present, some of it was carried down. In an experiment with 3 mgrms. Fe, 100 mgrms. Ti, and 100 mgrms. Zr as chlorides, no test for iron was obtained in the filtrate, thus showing that this amount of iron had been completely carried down. Also when 500 mgrms. alone (as $FeCl_3$) of Fe were present, some of the $Fe(OH)_3$ precipitated by NH_4OH did not dissolve in the H_2SO_3 solution, and even when ferrous iron was used, some $Fe(OH)_3$ sometimes precipitated during the heating on the water-bath. Moreover, the precipitation of titanium and zirconium was usually incomplete when much of these elements were present, more especially if the solution was not made distinctly alkaline with NH_4OH. This method is therefore a very unsatisfactory one.

G. D., Section 17: *Precipitation of Beryllium Phosphate as a Confirmatory Test.*—See B. E. Schlesinger (Thesis, Massachusetts Institute of Technology, 1902) Classen ("Ausgewählte Meth. d. Analyt. Chemie," 5th Ed., i., 715).

One mgrm. Be as $BeCl_2$ was in each of three experiments dissolved in water containing a little HCl; 1 cc. 10 per cent citric acid and 2 cc. 10 per cent ammonium phosphate solutions were added, the mixture was made strongly alkaline with ammonia, and it was boiled gently for about five minutes in a small flask; a small white crystalline precipitate separated. The experiment was twice repeated with 2 mgrms. Al as $AlCl_3$; no precipitate separated. The experiment was repeated with 0·5 mgrm. Be; no precipitate resulted (four experiments). Therefore this confirmatory test is hardly delicate enough, especially since beryllium has a very low atomic weight.

Test for Zirconium with Turmeric Paper.—The following procedure recommended by B. E. Schlesinger (Thesis, Massachusetts Institute of Technology, 1902) was tested:—"Dissolve the phosphate precipitate in HF (1 : 5), evaporate to one or two drops, and dip a piece of turmeric paper into it, heat the paper at 100° until dry." (Pink colour, presence of *zirconium*). In agreement with the results of Schlesinger, 1 mgrm. was found to be the limit of detectability. Even with 2 mgrms. the test was not a striking one. Moreover, titanium also caused a pink coloration of the turmeric paper, and the test was more delicate than in the case of zirconium. The blank test with strong HF was not perfectly satisfactory. The results obtained when HCl, instead of HF, was used were practically the same.

P. 51, N. 2: *Precipitation of Small amounts of various Elements by Ammonia.*—0·5 and 1 mgrm. of Al, Be, U, and Zr as nitrate, of Fe as $FeSO_4$, of Cr as $Cr_2(SO_4)_3$, of Ti as $TiCl_4$ were dissolved in separate experiments in 4 cc. HCl (1·12) and 30 cc. water in small conical flasks; the solutions were neutralised with NH_4OH (0·96), and about 1 cc. excess added; the mixtures were shaken several times, and then allowed to stand a few minutes; in each case the solution was transparent after the NH_4OH was added, but a distinct flocculent precipitate was observed after the shaking and standing. The precipitates were more difficult to see with chromium, aluminium, and uranium than with the other substances, but even in these cases 0·5 mgrm. was easily detected.

The experiments with aluminium, uranium, and chromium were repeated in a volume of 100 cc. containing 4 cc. HCl (1·12); 1 mgrm. Al was near the limit of detectability in a cold solution, but 0·5 mgrm. could be easily detected if the solution were heated nearly to boiling and allowed to stand. With uranium the limit of detectability was 1 to 2 mgrms. in a cold solution, and with chromium it was 2 mgrms. in a cold solution, but with 0·5 mgrm. Cr the solution became turbid on heating the NH_4OH solution to boiling.

Two mgrms. Cr as $KCr(SO_4)_2$ were dissolved in 4 cc. HCl (1·12) and a little water; the solution was boiled, diluted to 100 cc., cooled, and made alkaline with NH_4OH (0·96); a very small precipitate was seen after shaking and standing. This was filtered off, and the nearly colourless filtrate was heated to boiling; a finely divided light green precipitate then separated, showing that precipitation of $Cr(OH)_3$ by NH_4OH in the cold is incomplete.

Solubility of Zinc and Nickelous Hydroxides in Solutions containing NH_4Cl *and* NH_4OH.—To a number of solutions containing 500 mgrms. Zn as $ZnCl_2$, and varying amounts of HCl in 30 cc. of solution, known amounts of NH_4OH (0·96) were gradually added, 1·0 cc. HCl (1·12) being equivalent to about 1·5 cc. NH_4OH (0·96). The following table shows the amount of NH_4OH that had to be added (1) before a large precipitate was formed, (2) before a distinct odour of NH_4OH persisted after shaking, and (3) before the precipitate dissolved to give a colourless solution.

HCl (1·12) present. Cc.	NH_4OH (0·96) required. To give a large precipitate. Cc.	To give a distinct odour. Cc.	To dissolve the precipitate. Cc.
2	3	8	11
4	7	11	14
6	11	14	16
8	No precipitate	17	—

The experiment was repeated in a volume of 100 cc. containing 4 cc. HCl (1·12); the result was practically the same as in the corresponding experiment with an initial volume of 30 cc.

The experiments were repeated with 500 mgrms. Ni as $NiCl_2$, the initial volume being 30 cc.; the solution became blue when an excess of NH_4OH was added, but no precipitate separated even when only 2 cc. HCl were used. In an experiment with no acid, a large green precipitate resulted, which dissolved on adding about 6 cc. excess NH_4OH after the odour of NH_4OH was persistent.

Precipitation of Cobalt Hydroxide.—The foregoing experiments were repeated with 500 mgrms. $CoCl_2$ and 4 cc. HCl (1·12) in 30 cc.; when the NH_4OH was added slowly a moderately large green precipitate was always obtained, which did not dissolve on adding a large excess of NH_4OH; the supernatant solution was reddish brown. The experiments were repeated with 4 and 6 cc. HCl (1·12), the ammonia (3 to 4 cc. excess) being added all at once; no precipitate was obtained, but the solution was red coloured, and darkened on standing owing to oxidation.

The experiments were repeated in a volume of 100 cc. containing 4 cc. HCl (1·12); the results were the same, except that the amount of green precipitate formed was greater than in the smaller volume, and that a larger excess of NH_4OH was necessary, in order to prevent the separation of a precipitate when the excess of NH_4OH was added all at once.

(To be continued).

CORRESPONDENCE.

PLATINUM IN SO-CALLED PURE BORAX.

To the Editor of the Chemical News.

Sir,—In the Chemical News for August 28th (vol. xcviii., p. 104) there is a reprint of a paper by Mr. J. G. Rose, Government Analyst, Cape Town, dealing with the discovery of traces of platinum in so-called pure borax. I should like to occupy a few lines in pointing out a serious inconsistency, not in the facts as set forth in the paper, but with an argument drawn from them. Mr. Rose has made a very careful assay of borax, and "alleges" that he has found platinum. Granted; from the evidence of thorough work which the paper shows we may consider the case fairly proved. But from this fact Mr. Rose goes on to infer that the recent discovery of platinum in this district may be wholly or partly due to impure reagents. He says, "In view, however, of the recent *allegations* (Italics are mine) regarding the occurrence of platinum metals in South African rock formations, the matter assumes a somewhat different complexion."

May I just detail these "allegations" of the presence of platinum in South African rocks. In 1906 two Grahamstown gentlemen, Dr. Purvis and Mr. T. H. Harrison, "alleged" that they had found platiniferous rock. In February, 1907, in an analysis of a sample of similar rock by myself, I "alleged" the presence of both platinum and gold; this, I believe, was the first analysis actually published. Since then Messrs. Johnson, Matthey, & Co. of London have assayed *some hundreds* of samples of rocks from this district, and have "alleged" the presence of platinum in over 70 per cent of them, from a few grains up to 19 ozs. per ton. The Standard Bank of Johannesburg made several assays, and in one instance "alleged" 80 ozs., and in another 286 ozs. per ton. In addition to these "allegations" there are many independent analyses from chemists and assayers in Grahamstown, Johannesburg, Glasgow, Swansea, London, and elsewhere, all confirming the presence of the platinum metals; further, on several occasions actual metallic platinum has been seen in the rock, and beads of platinum from 50 to 98 per cent pure obtained by chemical means; the largest amount so far extracted was an ingot weighing 4 dwts. When assayed in London it was "alleged" to be 98 per cent pure platinum. The only chemists of repute who have so far consistently failed to find the metal are the Cape Colony Government analysts, but I think that the evidence of the "alleged" existence of platinum far outweighs their negative testimony. As for the presence of platinum in borax, that will have no bearing when the platinum is extracted with aqua regia, chlorine, or similar solvents.

Considering that fifty or sixty companies have been actually formed locally with a capital representing half-a-million pounds or more; that active negotiations are being carried on for the flotation of one or two of these on the London market; that other companies are doing their best to discover a commercial process for the extraction of the metal, it looks as if, in the Colony itself, the "allegations" are considered fairly proved.

To put the matter in a nutshell, if Mr. Rose's unsupported testimony is sufficient to establish the fact that Merck's borax contains platinum, why are the hundreds of analyses made by numerous competent authorities only "allegations" of the existence of platinum in certain South African rocks?—I am, &c.,

E. G. Bryant, B.A., B.Sc.

Grey Institute,
Port Elizabeth, South Africa,
September 19, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 10, September 7, 1908.

Fixation of Acetophenone by Benzoylacrylic Acid.—J. Bougault.—Boiling alkalis convert benzoylacrylic acid into glyoxalic acid and acetophenone, but if the acid is dissolved in a slight excess of alkali in the cold the reaction is different. On acidifying with acetic acid after twenty-four hours' contact a crystalline precipitate of diphenylacetic acid is obtained. The equation may be written—

$$2(C_6H_5—CO—CH=CH-CO_2H)+H_2O=$$
$$=CHO—CO_2H+(C_6H_5—CO—CH_2)_2=CH—CO_2H.$$

Probably the benzoylacrylic acid splits up into glyoxalic acid and acetophenone in the cold, but the change takes place more slowly than when heat is applied, and it is accompanied by the fixation of one molecule of acetophenone by one molecule of the undecomposed benzoylacrylic acid. Probably other acetones of the same type as acetophenone are capable of condensing with benzoylacrylic acid.

Nos. 11 and 12, September 14 and 21, 1908.

These two numbers contains no chemical matter.

MISCELLANEOUS.

Franco-British Exhibition, London, 1908.—It is officially notified that a Grand Prix has been awarded to the Wellcome Chemical Research Laboratories for their exhibit in Class 87 (Applied Chemistry and Pharmacy, &c.) at the Franco-British Exhibition.

Products from Milk-sugar and Calcium Hydroxide.—H. Kiliani.—The mixtures of milk-sugar and lime were freed from calcium salts of isosaccharine and metasaccharine, and from the mother-liquor all the calcium was removed by means of oxalic acid. The solution was then evaporated to a syrup, and extracted five times with absolute ether (Extract I.), four times with one part of absolute alcohol and two parts of dry ether (Extract II.), twice with a mixture of equal parts of the two solvents (Extract III.), and finally twice with absolute alcohol (Extract IV.). Each extraction was freed by distillation from the ether, and most of the alcohol then evaporated to a syrup and oxidised (II. and III. with concentrated nitric acid, IV. with the dilute acid). The first orientation experiments gave three oxidation products, *l*-tartaric acid, a tribasic acid $C_6H_8O_8$, perhaps identical with the acid of similar composition from isosaccharine, and a dibasic acid $C_6H_{10}O_7$, which differs from all known metameric acids.—*Berichte*, xli., No. 12.

NOTES AND QUERIES.

Ozokerite, Paraffin and Cerasin Waxes.—I should be obliged if any of your readers could tell me of any publication dealing with the chemical composition and analysis of ozokerite and paraffin and cerasin waxes.—A. Allender.

MEETINGS FOR THE WEEK.

Wednesday, 28th.—Society of Dyers and Colourists, 8. "Some Recent Improvements in Dyeing and Cleaning," by F. J. Farrell.

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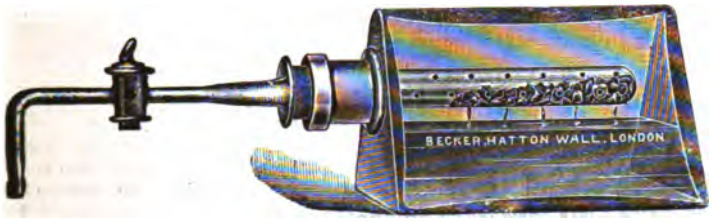

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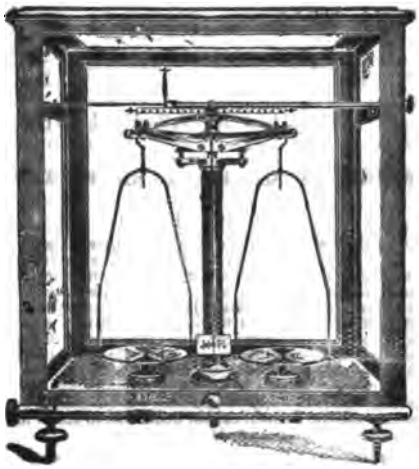

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A RAPID METHOD OF QUALITATIVE ANALYSIS.

By W. B. POLLARD.

Walton and Scholz (*American Chem. Journ.*, xxxix., No. 7) have lately shown the value of sodium peroxide as a reagent for opening up ores and other refractory substances, their method being to deflagrate the substances with this reagent and a metallic sulphide. Noyes in his system of qualitative analysis has also used sodium peroxide for decomposing those substances which have escaped attack by acids (*Tech. Quart.*, xvi., No. 2), and for the separation of the metals of the ammonium sulphide group (*Journ. Am. Chem. Soc.*, xxx., No. 4).

The ease and rapidity with which substances are decomposed when deflagrated with sodium peroxide, together with the fact that when the resulting melt is dissolved in water and filtered, a sharp separation of the elements into two groups occurs, has suggested that this treatment might be made the basis of a system of qualitative analysis. The subdivision of each of the groups thus obtained might then be accomplished by means of the ordinary group reagents.

The decomposition of the substance by alkali instead of acid is therefore the essential characteristic of the method.

The advantages of this procedure may be stated as follows:—

1. The method can be applied to practically all substances.
2. Large quantities can be opened up with as much ease as small amounts.
3. All organic matter is destroyed.
4. The group separations are always done under standard conditions.
5. A great economy of time is effected.

It may be argued that a substance like hæmatite is not completely attacked when deflagrated with sodium peroxide. This would be fatal in quantitative work, but is not a serious drawback when we only require to know what elements are present in the substance; and, moreover, the process can be repeated on the undecomposed portion if desired. The fact that silicates, which constitute the chief portion of the earth's crust, are quite easy to examine in this way, but require special treatment when the ordinary system is followed, is a point strongly in favour of some such modification as the above.

It may also be said that if the substance is not first dissolved in some solvent, much useful information concerning its nature is lost. It seems more systematic, however, to find out first what elements are present and then to ascertain how they are combined, and to reserve this test till the end of the analysis.

As sodium peroxide attacks all materials it is necessary to arrange the reaction in such a way that metallic impurities are not introduced.

This can be done by making a mixture of approximately one part of the substance, in the state of finest powder, with one part of vaseline and five parts of sodium peroxide, and igniting it on a thick iron plate, or in the cavity of a scorifying mould by means of a match or Bunsen burner. If the metal support is thick the heat is conducted away before the surface can become attacked. The excess of vaseline used also acts as a protection to the metal. The substance under examination should be dry, and the sodium peroxide used should be quite fresh, as samples which have been exposed to the air deflagrate very badly. Too much vaseline also should not be used, as the energy of the reaction is thereby considerably lessened. As a result of the action of the sodium peroxide, every element now exists in a definite condition in the melt, which is independent of the way in which it was formerly combined.

The melt is now dissolved in water and filtered, when a sharp separation of most of the elements into two groups is obtained. The soluble portion contains the soda salts of the metallic and non-metallic acids, for the most part in their higher states of oxidation, together with those oxides of the metals which are soluble in an alkaline solution. The insoluble portion contains the oxides, peroxides, and carbonates of the metals, any insoluble soda salts, and possibly a portion of the substance which may have escaped attack. A few elements like lead will be found in both portions, but this occasions no difficulty.

It must be noticed, however, that the results obtained by extracting the melt with water are not always the same as those obtained when a solution of the metal is precipitated with sodium peroxide. For example, on extracting the melt, titanium finds its way into the soluble portion, and small amounts of iron also dissolve under certain conditions.

Each of these portions can now be tested with the usual group reagents, but as the groups are smaller the recognition of the individual members becomes easier.

On dissolving the insoluble portion in acids, a little of the undecomposed substance may remain, together with the excess of vaseline, but these may be filtered out. A little phosphoric acid, which has not been rendered soluble by the action of the sodium peroxide, may also be present in the solution.

Instead of testing the soluble portion of the melt with the group reagents in the usual way, the following may often be sufficient:—The solution is first acidified with hydrochloric acid, when the colours due to the formation of perchromic acid and titanium trioxide appear. The solution may then be neutralised with excess of soda, a little sodium sulphide added, and then boiled. On filtering out the metals precipitated by the sulphide, the solution may be again acidified with hydrochloric acid, and the sulphides of those metals, which form thio-salts, filtered out. A little silica may separate here, but this does not in any way interfere with the examination of the sulphides.

After oxidising the filtrate, aluminium may be precipitated by boiling with excess of sodium acetate and a little sodium phosphate. Potassium can then be precipitated in the filtrate as cobalti-nitrite. As, however, potassium is sometimes present as an impurity in sodium peroxide, this fact must not be overlooked when testing for that element.

Sodium must, of course, be looked for in the original substance, and mercury should also be tested for separately on account of its volatile nature.

In examining ores and minerals in the field, it is seldom necessary to test for more than two or three elements; the results, however, must be obtained quickly, and with the aid of a very limited supply of apparatus; in cases such as these the method may possibly prove of use.

A METHOD OF ANALYSING SHELLAC.*

By PARKER C. McILHINEY.

The method of analysis which is in most common use at the present time, both in England and in the United States, for the determination of the amount of rosin in shellac, is an indirect method depending upon the different powers of a shellac and of rosin to absorb iodine from a suitable solution. Different operators prefer different methods of making this test, some preferring to use the old Hubl method, and others the more modern Wijs method, as modified by Langmuir. Either of these methods is capable of giving reasonably satisfactory results, although the Langmuir method is certainly much to be preferred, both on the score

* Read before the New York Section, March 6, 1908. From the *Journal of the American Chemical Society*, xxx., No. 5.

of accuracy and of speed. Another method which is in use is that proposed by Parry, depending upon the solubility of the resinate of silver made from common rosin, in ether, while the corresponding resinates from shellac are insoluble. This method labours under several disadvantages and sources of error, of which the two principal ones are the solubility of the unsaponified portion of the shellac in ether, and the danger of a decomposition of the resinate of silver before it can be separated and determined.

A direct method of separating shellac and rosin and recovering the rosin, at least in a substantially unchanged form, is greatly to be desired, and several experimenters have attempted to make such a separation by taking advantage of the solubility of rosin in petroleum ether, a solvent in which shellac is insoluble. No method of extracting from even a finely pulverised sample of shellac the portion soluble in petroleum ether seems to be capable of removing more than a small part of the rosin contained in the sample.

Shellac dissolves in alcohol, except for the wax contained in it; petroleum ether dissolves to some extent in alcohol, and it was thought that by first dissolving the shellac to be analysed in alcohol, then adding to the solution all the petroleum ether which it would dissolve, and then adding water, so as to so dilute the alcohol that it would no longer have any material solvent power upon either rosin or shellac, there should result a separation of the dissolved petrolic ether containing in solution the rosin and the wax, but free from the resinous constituents of the shellac. Upon trying this process, it appeared that it was correct in principle, but that on account of the limited solubility of petroleum ether in alcohol, the separation was not quite complete. Upon substituting for ordinary alcohol, glacial acetic acid, or absolute alcohol, in which the sample of shellac to be examined is dissolved, and then adding to it petroleum ether, which is miscible in all proportions with acetic acid, or absolute alcohol, it was found practicable to combine with the rosin and the wax so large an amount of petroleum ether, that upon adding water, an almost complete separation of the rosin and wax from the resinous part of the shellac could be effected.

Based upon these facts, the following process was devised:—Dissolve in 20 cc. of glacial acetic acid (about 99 per cent), or the same volume of absolute alcohol, 2 grms. of the sample to be analysed. This requires a gentle heat. Add to the solution, after cooling, 100 to 300 cc. of petroleum ether, boiling under 80°. This addition of petroleum ether should be made slowly, because the addition of so large an amount of petroleum ether precipitates from its solution a part of the shellac, combined, apparently, in case acetic acid was the original solvent, with acetic acid to form a liquid precipitate. It is manifestly desirable that this precipitation of part of the shellac should not be effected by too sudden an addition of petroleum ether, as it might then contain some rosin carried down mechanically with it.

We now have a solution containing both petroleum ether and glacial acetic acid, or absolute alcohol, and containing in it, in solution, all the rosin, all the wax, and most of the resinous part of the shellac. Add now to this solution, drop by drop, 100 cc. of water, agitating the liquid during the addition. The water "unites with" the alcohol or acetic acid, and separates from the liquid the petroleum ether with whatever is soluble in petrolic ether. This includes the rosin and the wax. The shellac is also precipitated, but as it is insoluble in petroleum ether it remains as a precipitate suspended in the diluted alcohol or acetic acid. The separation of the two liquids takes place very rapidly, and it is an easy matter to effect a complete separation of the two layers in a separating funnel. The petroleum ether layer is washed once or twice with water, and then filtered through a dry paper into a weighed flask, from which the petroleum ether is then distilled off, leaving the residue of rosin mixed with wax, which is weighed. To the weighed residue neutral alcohol is added, and the flask heated to dissolve the rosin in the alcohol. The liquid is then titrated with N/5 or N/10 caustic potash. This gives a measure of the amount of rosin present, as the average combining equivalent of rosin does not vary greatly from 346. This is the figure used for this titration in the Twitchell process. It is much better, however, not to depend upon his determination of the rosin, but to proceed to an actual separation by adding to the neutralised alcoholic solution a distinct excess of alkali and a sufficient quantity of petroleum ether; the mixture is then transferred to a separating funnel, thoroughly agitated, and some water added. The liquids are then separated, the petroleum ether layer being washed with water, and the alcoholic solution of the rosin extracted once more with petroleum ether. The petroleum ether solutions are then united, the petroleum ether distilled off, and the residue of wax weighed. The alcoholic solution containing the rosin is then boiled until the alcohol has been expelled, and, if necessary, a further addition of water made; then dilute hydrochloric acid is added, so as to precipitate the rosin. This should be done in a weighed flask; the acidified liquid can now be decanted off through a wet filter-paper, so as to leave behind in the flask the major part of the rosin, which is washed with water. The remainder of the rosin upon the filter-paper after sufficient washing, is extracted with petroleum ether back into the weighed flask from which the water has been decanted as perfectly as possible. Upon distilling off the petroleum ether, the rosin is left behind in a condition to weigh. If the whole of the last traces of water have not been removed by the distillation with petroleum ether, it is well to add a further quantity of dry petroleum ether and distil again.

Attempts were made to use other solvents, particularly coal-tar benzene instead of petroleum ether. It was found, however, that benzene dissolved considerably more out of the shellac than the wax.

If it is desired to determine only the rosin in the shellac, it is unnecessary to distil off all the petroleum ether from the solution containing the wax and the rosin. This solution may, instead, be treated directly with an alkali capable of combining with the rosin, while leaving the wax unattacked. The most convenient method which I have found for accomplishing this is to add to the petroleum ether solution, after filtering it to remove from it anything which it may contain in suspension, a solution of sodium hydroxide in absolute alcohol. Such a solution I have found it convenient to make by dissolving in ordinary 95 per cent alcohol the appropriate amount of metallic sodium. Such a solution, when added to the petroleum ether solution, mixes perfectly with it, and the alkali combines with the rosin, and the mixed solution allows the saponified rosin to be extracted from it by agitation with water, or better, with slightly diluted alcohol. Instead of using a solution of sodium hydroxide in absolute alcohol, it should be possible to use the method of separating rosin from neutral substances in petroleum ether solution, that is used in the Twitchell process of determining rosin in admixture with fatty acid. This consists in agitating the petroleum ether solution with an alkaline aqueous solutions containing some alcohol, made by dissolving 1 grm. of potassium hydroxide with 10 cc. of alcohol in water, and diluting to 100 cc. I have found the other method of procedure advantageous, however, and believe it to give a more exact separation. The solution drawn off from the petroleum ether, after extraction by alkali, and containing in solution the rosin, combined with potassium or sodium, together with whatever alcohol has been used to promote the combination and to facilitate the mechanical separation of the two liquids, is heated for some time to remove the alcohol, and is then acidified with hydrochloric or with sulphuric acid to precipitate the rosin. The precipitated rosin is then weighed as before.

Without entering into a discussion of the exact chemical composition of shellac wax, it may be proper to state that the wax may be separated commercially from shellac by two essentially different methods, which will probably give waxes having somewhat different compositions. If the

shellac is dissolved in an alkaline solution and the wax which remains in suspension is filtered out, it is probable that different results will be obtained from what would be obtained by the other process, consisting of a solution of the shellac in alcohol, and filtration of the solution to remove the wax which will remain in suspension. It is to be expected that these two processes of solution would leave, in the undissolved wax, small amounts of ingredients of different characters, and, furthermore, the alcohol used in the second process would dissolve small amounts of the wax which would probably be quite insoluble in an alkaline aqueous solution. Again, it is quite probable that a caustic solution would behave towards shellac wax somewhat differently from a dilute carbonate solution. For the sake of uniformity and simplicity, I am assuming that shellac wax is quite free, when pure, from all acid substances capable of uniting with free alkali. It is nevertheless true that the user of shellac who wishes to have a determination made of the amount of wax which it contains, usually desires this information in order that he may know how much of the shellac will remain undissolved in alcohol. This amount of insoluble wax will, of course, vary to some extent, depending upon the strength of the varnish that he prepares with it; that is to say, the number of pounds of shellac which he uses to a gallon of alcohol. As the analyst can hardly be expected to take account of this in making his test, it appears more rational to regard as wax the whole of those matters contained in the shellac that remain insoluble in an alkaline solution.

In making a determination of the amount of wax in a sample of shellac by this method, it is to be observed that some constituent of shellac wax is evidently only difficultly soluble in petroleum ether. Upon submitting several portions of the same lot of shellac to analysis in this way, the only difference made in the different determinations being that varying amounts of petroleum ether were used, the series of analyses gave, with increasing amounts of petroleum ether, increasing percentages of wax, until the proportion of about 125 to 150 cc. of the solvent to 1 grm. of a shellac containing about 5 per cent of wax was reached. It may be that by using some other solvent such as benzene for the determination of wax in this way, a smaller amount will suffice, but as it seldom happens that only wax without rosin is to be determined, petroleum ether is the most generally applicable solvent. The solubility of rosin in petroleum ether is so easy and complete that no difficulty is experienced in extracting from 2 grms. of shellace 50 per cent of rosin, using 100 cc. of petroleum ether.

Whenever in the course of an analysis by this method a quantity of material is separated by acidifying the aqueous solution which should contain the rosin, its identity may be established with some certainty by determining its iodine figure by the Langmuir method, and its acidity by titrating with alkali in alcoholic solution.

The petroleum ether referred to here is a solvent made by re-distilling 71° Be benzine, separating for use that part which distils below 80°. This fraction constitutes a large proportion of 71° benzine, and such a re-distillation gives, at a comparatively small cost, a satisfactory solvent for the purpose. If a determination of only the rosin is desired, it is, of course, unnecessary to be very particular as to the volatility of the solvent used, as it is unnecessary in such case to distil it off before extracting the rosin by alkali.

By treating pure shellac according to this process, it is possible to separate from it a small amount, sometimes as much as 1 per cent, of materials soluble in petroleum ether. This small amount of resinous matter, when examined, proves to be something essentially different from common rosin. Its odour and its low iodine figure indicate that it is some resinous constituent of shellac, perhaps a small amount of the major constituent, which is slightly soluble in petroleum ether. As it is difficult to imagine that by this process rosin if a normal constituent of pure shellac would escape detection, and as the small amount of resinous matter here obtained is essentially different from rosin, it is reasonable to conclude that, contrary to the idea held by many, common rosin or a material similar to it, is not a natural constituent of pure shellac, but that any rosin or colophony, which can be separated in a state of reasonable purity from the sample of shellac, was originally added to the shellac as an adulterant.

The process here described allows the analyst to separate in a form convenient for exhibition either as evidence in court or as an ocular demonstration for his client, any rosin which may have been added as an adulterant to shellac.

Shellac varnishes may contain beside true shellac not only rosin, but other gums and resins soluble in alcohol. It becomes therefore a matter of interest to ascertain how some of these other resins behave when treated by this process. Two samples of manilla, when treated, using absolute alcohol as the first solvent, gave, respectively, 41·2 and 43·3 per cent of matter soluble in petroleum ether. The acidity of these two lots of matter soluble in petroleum ether was in the case of the first sample such that 1 cc. of normal alkali neutralised 411·7 mgrms., and in the case of the second 470·7 mgrms. Two samples of Kauri gave, respectively, 37·9 and 27·0 per cent. Upon titrating with standard alkali these portions soluble in petroleum ether, it appeared that 1 cc. of normal alkali was capable of neutralising 903·6 mgrms. and 742·5 mgrms. respectively. Of Sandarac, two samples, when similarly analysed, gave 34·96 and 36·19 per cent, having such an acidity that of the first, 541·2 mgrms. would neutralise 1 cc. normal alkali, and of the second 552·5 mgrms. would neutralise 1 cc. Of Dammar, 89·9 per cent proved to be soluble, while the resin of *Shorea roburta*, a sample of which was kindly sent by Mr. W. Risdon Criper, of Calcutta, gave 69·5 per cent of soluble matter.

A number of attempts were made to effect a satisfactory separation of the wax before separating the rosin from the shellac. It was found, however, that on account of the solubility of wax in alcohol and in glacial acetic acid, this separation could not well be made by filtering out the wax before the addition of petroleum ether. Neither were attempts which were made to separate the wax by a preliminary solution of the shellac in aqueous alkali successful in furnishing a method that at all approached in feasibility to the method already described.

THE WAVE THEORY OF MOTION.

By JOHN McDOWALL.

In the Chemical News, vol. xcviii., p. 61, I published a statement to the effect that motion could only be transmitted through the agency of a conducting medium.

The data on which this statement was based showed that the fundamental unit of matter was a wave of some kind, though it did not give any information as to the properties of the wave with respect to the velocity at which it was propagated through space. There were two possibilities, either the wave passed through space with a constant velocity which would be the normal wave, or else it ran at a speed proportional to its amplitude which would be abnormal.

I tried at first to get the normal kind of wave to fit in with the observed properties of the motion of matter, but failed to get a good explanation on those lines, and had to make use of a wave which ran at various speeds depending on its compression, as it seemed possible that the unique properties of the ether might make such a wave possible.

I have recently resumed work on the wave which runs at constant speed, and have overcome the difficulties which made me set it aside as impracticable before.

I shall put it before the reader in the same order as it was evolved so as to show why I abandoned it and how the difficulties were surmounted.

If we imagine two spheres to be passing through space

at a constant velocity in such a way that the paths which they make in space are parallel to each other, then they are relatively at rest with respect to each other; if we imagine the direction to be slightly changed, the sphere on the left being made to move towards the right, and the sphere on the right being made to move towards the left, then this would produce relative motion which would vary depending on the angle which the paths which the two spheres described in space made when produced to meet; the greater this angle the higher would be the relative velocity although the speed at which the spheres moved was constant.

Now it seemed that in this simple principle I had the key to the situation. Our ideas of motion are all of a relative nature, we know nothing of the absolute motion of matter in space. Any motion of the nature of a general drift of matter in space would be unknown to us because we would have no object absolutely at rest by which we could judge it; in fact, the motion common to all matter would be unknown to us, we can only recognise it when the motion is different in direction and velocity.

It would perhaps be best to introduce here the idea of the absolute resultant of the fundamental unit.

The absolute resultant of the fundamental unit is obtained by compounding any motion it has in the atom with the motion of the atom when agitated by heat, and the resultant of this with the motion of the earth round its axis, and the resultant of this with the motion of the earth round the sun, and the resultant of this with any drift which is common to the whole of matter, and on account of which we cannot recognise it, is most lucidly conceived as the imaginary line traced out by the fundamental unit in the ether as it passes through space and includes all and every motion that influences the fundamental unit. This is called the absolute resultant of the fundamental unit.

Let us return to the two spheres which can only move at constant speed, and instead of placing them so that the paths they describe are parallel to each other we place them behind each other so that there is a space between. Now, if the spheres were made out of matter whose fundamental units were waves which moved at constant speed, then when placed behind each other in this fashion they would not be able to vary the distance between them; if it was possible to give one an impetus it would only increase the amplitude of the fundamental wave, it would not make it move faster thongh it would take more force to stop it.

Now, instead of working with two spheres it will be convenient to work with a finite number, say ten or fifteen.

Suppose we consider a plane of two dimensions in which those spheres move, one dimension is devoted to the constant speed, the other to the relative speed.

Now, the only figure in which the spheres could move relatively and still remain in the region of collision would be represented by a line (in all those conditions it is necessary to consider the speed which is constant to be very much greater than the relative speed); if one sphere was placed out of line it could not collide with its neighbours.

Now, suppose we introduce another dimension, then the figure in which relative motion would be possible and the spheres be still within the region of collision would be represented by a plane. The spheres would be arranged on a plane which would move at right angles to its surface at constant speed, which must be very high compared to the relative motion of the spheres in the plane towards each other. Of course this relative motion will vary in velocity, depending on the inclination of the path of one sphere to that of a neighbour. This plane is important to remember.

Now, suppose we introduce another dimension yet, then the spheres would need to move with constant velocity in a certain direction, and over and above this there would be three dimensions in which varying inclinations of the path of the sphere to its neighbours would show up as varying relative velocity.

Now, we conclude from this that if this principle of constant speed and varying relative velocity is to be applied to explain the properties of motion in matter, then the whole bulk of matter must be speeding along a fourth dimension with constant velocity, which, if the rigidity of the ether goes for anything, must be very high—enormous, in fact—and that a minute deviation in the direction of the absolute resultant would show up as brisk relative motion, and a greater deviation as violent motion in the remaining three dimensions.

Now the question arises, if this dimension exists why do we not become aware of it? And this is what gave me much difficulty for a long time. If a man who was colour blind and could not see the red end of the spectrum was not told there was such a thing as red light, he would never be able to think that such a thing existed. We gain our knowledge of our surroundings by experience. Now, if we had a dimension in which we could not move, we would never be able to experience motion in it and would not be able to conceive it.

The difficulty is that there is a species of relative motion, which was the reason that I abandoned the theory at first, but on further investigation is one of the chief supports.

To go back to the standard plane on which the analogy is based.

We have the plane moving at constant speed in a direction at right angles to its surface. Now, if one of the spheres was to have an excess of relative motion its path would be more sinuous than the others, it would contain the same length for equal times but would contain less linear length of the dimension in which the plane moved with constant speed, and it would consequently drop out of line and be removed beyond the region of collision.

Now, the same thing should result with an additional dimension; that is to say, that a piece of matter which had a very high relative velocity should tend to drop behind matter with less, and should be removed beyond the region of collision. If the piece of matter had motion in its atoms, and fundamental units, then it would be removed gradually, for a unit whose motion reinforced the relative motion of the piece of matter would be left behind first, whereas a unit whose motion was opposite to the general motion of the piece of matter would take longer to be thrown behind. In fact, a body with excessively high relative velocity should leave a train of units behind it and gradually grow less in mass, and finally disappear in a mysterious fashion beyond the region of collision.

Now, there is no evidence to show that this happens; it is indeed a fact that it does not happen, for which reason, combined with the fact that the relative motion which would take place at high speeds was not observed, made me abandon this explanation and substitute the wave which moved with a velocity proportional to its compression, as that was the only kind of wave which would suit.

Now I have returned to the wave which runs at constant velocity with more success.

We have seen that relative motion must occur in this extra dimension to an extent depending on the relative velocity. This makes it possible for us to become aware of it. On the other hand, this extra dimension is an abnormal dimension. In the first place it contains the great bulk of the motion of matter. Our attitude towards it is, so to speak, "end on," whereas we are broadside on towards the other dimensions; relative motion in it can only be produced by producing an excess of relative motion in the ordinary dimension.

All those things show that this dimension is abnormal, so that it seemed possible that it would appear different to us than the other dimensions whose properties were all the same. It might, in fact, be well known to us, but we might look on it as something else. The next thing to do was to look for some principle which was of a fundamental nature, and which might be the extra dimension in disguise.

It should be closely associated with ordinary space and

also motion, and might possibly show the disturbe reflection of a dimension in it.

Now the only principle which seems to meet those conconditions is time. Time must clearly have an objective reality; the fact that we can measure it shows this. Time has one dimension. We speak of a length of time, and a space of time, and although our conception of this is different from our conception of a length of space the abnormal properties of matter might explain this.

In order to test this we have to see exactly the relations of time to motion and to ordinary space, and then see if those same relations hold for the extra dimension.

Suppose we measure off a finite length of an ordinary dimension, say, thirty metres; now suppose we send two projectiles over this space, one projectile being a bullet from a gun, the other being thrown by hand.

Both those projectiles would do the same act with respect to the space—they would both move over it, but while doing this they would differ on two points. The bullet would contain a much larger quantity of relative motion than the other projectile, and would also consume a smaller length of time.

Now, how would the fourth dimensions act under those same conditions? The result of superposing on the absolute resultant different quantities of relative motion is to make it more oblique; with respect to the fourth dimension it moves slower along it and consumes less of it than if a smaller quantity of relative motion had been used. This shows there is a connection, but does not establish the identity of the two.

This may be done as follows:—Suppose two men had to meet at a certain place, what are the mechanical conditions which such a meeting involves? From an absolute point of view the two men would meet when their absolute resultants intersected, both men being at the point of intersection.

Suppose we do not take the fourth dimension into account, simply fixing a point in the ordinary dimensions, then both men would do the act of passing over the space to this point; perhaps one would pass an hour after the other, and they would not meet. This shows that to fix their absolute resultants rigidly another condition is needed; by simply fixing the point of meeting in the ordinary dimensions there is a degree of freedom, and this degree of freedom is along the fourth dimension. It is therefore obvious that if we have to make their meeting a mechanical certainty that we have to fix them in respect to this dimension. Also when this is done their absolute resultants as far as they concern each other are rigidly fixed, and meeting is a mechanical certainty.

We have, in fact, to make them on a level in the fourth dimension, and also make them pass over the ordinary dimension to the point of meeting. When those conditions are attended to then they will meet, the absolute resultant being rigidly fixed in space.

Now if they are not on the same level in the fourth dimension, how are they to manage to get level?

Let us go back to the standard plane on which the analogy is based. We have the plane moving at constant speed. Let us imagine one sphere to be placed in front of it moving at the same speed, and another to be placed behind, how are those two spheres to get into the plane?

The one in front can do so by pursuing a sinuous or oblique path, which means that it lets out a large quantity of relative motion, and enables the plane to draw up on it; the sphere behind would do the opposite, describing a path containing less relative motion than the average relative motion of the spheres in the plane, and would catch up on it. Now, the men in order to get on a level in the fourth dimension must do the same thing while proceeding to the appointed place; if they are not on a level one of them must let loose a large amount of relative motion and the other a small amount, both doing the required act with respect to the ordinary dimensions by moving across the space to the point of meeting. Now the means by which they know if they are on a level is by means of a watch, and the one who is late produces a large amount of relative motion, and the one who is early a small amount, doing this to the proper extent, and they meet.

Two bodies are removed as securely from the region of collision if they cross the same point at different times as if they were in a different part of space.

Our minds are constructed to think and conceive this fourth dimension as time, and the other three as space, because the properties of the motion of matter to this dimension is different to the others, and therefore we conceive it in a different way so as to accommodate ourselves to the different properties of motion towards it.

There is another thing I should like to note before concluding this short statement, and that is the property of projectiles or other bodies to be as a system in a state of relative rest to an object a long distance away if they described enclosed curves.

If we had the two men at a distance from each other in a state of relative rest, then if one of them described small ellipses, no matter how much relative motion he let loose he would still be in a state of relative rest; as a system when the axes of the ellipse was very short compared with the distance of the other man if he had let loose the same amount of motion in a straight line, he would be in a different position to the other man, not only with respect to the ordinary dimensions but also to the fourth dimension—time.

Now the reason that he is in a state of relative rest while describing an enclosed figure is because he undoes in one part of the curve what he does in the other.

This absolute resultant, if he was in a state of relative rest, is in a certain direction; as he begins to move in an ellipse when he moves away from the observer he superposes on this resultant a component which makes it more oblique along the fourth dimension; on the return he superposes a component which makes the absolute resultant abnormally straight along the fourth dimension; so we have simply positive and negative accelerations in the fourth dimension which balance each other, and the body as a whole is at relative rest, so we can bottle up relative motion by making the units of matter describe enclosed curves, and, of course, the same thing holds for the planes and other bodies which as systems are at relative rest.

Sebaieh, Upper Egypt.

THE INFLUENCE OF FINE GRINDING ON THE WATER AND FERROUS-IRON CONTENT OF MINERALS AND ROCKS.*

By W. F. HILLEBRAND.

(Concluded from p. 206).

Effect of Grinding in Air.

THE table shows in the first place with respect to the moisture, that while some rocks and minerals contain in the lump, and even in material as fine as 100-, or even 120- and sometimes 150-, mesh size, little moisture (2*a*, 5*a*, 12*a*, 13*a*) or none at all (14*a*, 15*a*), many, and particularly those that have undergone more or less alteration into hydrous minerals, contain in coarse condition a good deal of loosely held water (3*a*, 4*a*, 6*a*, 8*a*, 9*a*). Since this water is characteristic of certain minerals and affords a valuable index of the character and extent of alteration or of the presence of zeolitic minerals, it should not be excluded from the tabulated analysis, as Mauzelius recommends. On the other hand, the petrologist reports "kaolin" as the chief hydrous mineral in 13. The analysis shows absence of loosely held water, therefore the mineral is probably in the crystallised form of kaolinite, which does not lose water at 100°. Since, further, such powders of grain as coarse as 100-, 120-, and even 150-mesh often show much

* From the *Journal of the American Chemical Society*, xxx., No. 7.

No.	Material.	Maximum grain of bulk sample.	Exp. No.	Conditions of grinding. Grms. of bulk sample.	Enveloping medium.	Time, minutes.	H_2O per cent. 105°−.	H_2O per cent. 105°+.	FeO per cent, corrected for H_2O taken up. Pratt method.		Cooke method.	
1.	Basalt*	0·08 mm. < 150-mesh	*a*	Unground	—	—	0·24	—	7·03	7·07	—	—
			b	4	Absolute alcohol	30	0·34	—	7·09	7·11	—	—
									7·09	7·12	—	—
			c	4	Air	30	0·32	—	7·14	—	—	—
									7·01	7·07	—	—
									7·08	7·16	—	—
			d†	20 ?	Air	?	0·45	—	7·18	7·23	—	—
			e	4	Air	120	0·83	—	7·12	—	—	—
									6·87	6·93	—	—
2.	Diorite‡	0·08 mm. < 150-mesh	*a*	Unground	—	—	0·07	—	3·65	3·65	3·55	3·52
			b	4	Absolute alcohol	120	0·32	—	3·48	3·50	3·41	3·50
			c	4	CCl_4	120	0·48	—	3·30	3·37	3·26	3·31
			d	4	H_2O	140	0·53	—	3·41	3·44	3·37	3·39
			e	4	Air	120	0·54	—	3·29	3·37	3·30	3·39
									3·41	—	—	—
			f†	20 ?	Air	?	0·28	—	3·52	—	3·37	—
3.	Andesite	0·08 mm. < 150-mesh	*a*	Unground	—	—	1·14	—	2·57	2·58	—	—
			b	4	Absolute alcohol	15	1·21	—	2·58	—	—	—
			c	4	H_2O	15	1·39	—	2·53	—	—	—
			d	4	Air	15	1·46	—	2·53	—	—	—
4.	Andesite	0·08 mm. < 150-mesh	*a*	Unground	—	—	1·01	3·40	3·32	3·34	—	—
			b	4	Air	30	1·18	3·54	3·09	—	—	—
5.	Diabasic norite or gabbro§	0·08 mm. < 150-mesh	*a*	Unground	—	—	0·03	0·66	13·55	13·35	13·54	13·24+
			b	4	Absolute alcohol	20	0·10	0·86	13·04	13·14	13·11	—
									13·15	13·20	—	—
									13·23	—	—	—
			c	4	CCl_4	60	0·34	0·94	12·95	13·00	—	—
			d	4	Air	30	0·10	0·66	12·91	12·93	13·06	13·07
									13·06	13·07	—	—
			e†	20 ?	Air	?	0·10	0·65	—	—	—	—
			f‖	4	Air dried by H_2SO_4	120	0·74	1·00	12·72	12·79	—	—
6.	Diorite-porphyry . .	0·18 mm. < 100-mesh	*a*	Unground	—	—	0·29	1·97	2·71	2·72	—	—
			b	20 ?	Air	?	0·42	2·07	—	—	2·38	—
			c	4	Air, machine A	120	0·91	2·26	2·30	2·31	—	—
			d	4	Air, machine B	120	1·90	2·73	1·54	1·54	—	—
			e‖	4	Air-dried by H_2SO_4, machine B	120	1·36	2·38	1·80	—	—	—
7.	Altered basaltic greenstone	0·18 mm. < 100-mesh	*a*	Unground	—	—	0·22	3·13	6·68	—	—	—
			b	4	Air	120 or more	1·70	4·19	3·74	—	—	—
8.	Quartz-diorite	0·11 mm. < 120-mesh	*a*	Unground	—	—	0·37	2·03	2·58	—	—	—
			b	4	Air	120 or more	1·08	2·33	1·69	—	—	—

9. Quartz-diorite	0·11 mm. < 120-mesh	*a*	Unground	—	—	0·35	1·60 1·64	2·08	2·09	—	—
		b	4	Absolute alcohol	120	0·54	1·80	2·02	2·02	—	—
		c	4	Air	30	0·40	1·56 1·64	2·04	2·07	—	—
		d	4	Air	120	1·01	1·86 1·95	1·48	1·48	—	—
10. Quartz-diorite	0·11 mm. < 120-mesh	*a*	Unground	—	—	0·21 0·22	0·65 0·65	2·55	2·58	—	—
		b	4	Absolute alcohol	120	0·39	0·88	2·58	2·58	—	—
		c	4	CCl_4	120	0·41	0·80	2·47	2·51	—	—
		d	4	Air	120	0·69 0·70	1·05	2·33	2·34	—	—
11. Granite	0·11 mm. < 120-mesh	*a*	Unground	—	—	0·12	0·46	2·28	2·28	—	—
		b	20?	Air	120 or more	0·21	0·53	1·85	—	—	—
12. Granite	0·11 mm. < 120-mesh	*a*	Unground	—	—	0·03	0·24	1·93 2·08	2·01 —	—	—
		b	4	Absolute alcohol	120	0·32	0·64	2·01 2·06	2·03 —	—	—
		c	4	Air	120 or more	0·67	0·72	1·86	1·86	—	—
13. Altered basaltic rock..	0·18 mm. < 100-mesh	*a*	Unground	—	—	0·03	2·00 2·12 2·15	8·33	8·34	—	—
		b	4	Absolute alcohol	120	0·54	2·42	8·18	8·21	—	—
		c	4	Air	30	0·15	2·19	8·24	—	—	—
		d	20?	Air	Not known, but very long.	1·16 1·21	3·34 3·36	5·86	—	—	—
14. Quartz	Mostly finer than 0·08 mm. < 150-mesh	*a*	Unground	—	—	None	0·06¶	—	—	—	—
		b	4	Air	120	0·35	0·45	—	—	—	—
15. Berlin porcelain, unglazed**	?	*a*	Uncrushed	—	—	None	None	—	—	—	—
		b	?	Air	?	0·62	0·18	—	—	—	—
16. Meerschaum††	Coarse	*a*	Unground	—	—	5·76	6·79	—	—	—	—
		b	4	Air	600	6·33	9·81	—	—	—	—

* The rather wide variations for FeO in the same series, particularly *c*, are ascribed to the refractoriness of an iron-bearing constituent, which necessitated several re-treatments.

† Sample originally analysed.

‡ Difficult to decompose by HF in powder coarser than 150-mesh.

§ An iron-bearing garnet in this rock was extremely refractory unless very finely ground.

‖ Exposed to air of room after grinding.

¶ H_2O 105°+, by ignition.

** Also 0·80 per cent H_3O as total ignition loss.

†† Tests made in 1902.

less than 0·1 per cent moisture, if hydrous alteration products are absent, it is reasonable to conclude that there has been occasioned relatively as little oxidation of iron in such powders as absorption of moisture, on the assumption that the small amount of water found results from the increased surface exposure due to crushing, and is not inherent. Therefore, if the powder is completely decomposed by hydrofluoric acid, it is safe to assume that a determination of ferrous oxide in it, carried out with proper precautions, will give a result close to the truth.

It is further evident that, whereas long grinding in air (two hours or more) by the mechanical grinder in most cases gives rise to marked oxidation of iron (6*d*, 7, 8, 9*d*, 11, 13*d*), and increase in the water content (6*d*, 7, 8, 9*d*, 10*d*, 13*d*, 14) these effects are usually not strongly marked when the grinding was by hand for only thirty minutes or less (5*d*, 9*c*, 13*c*, and especially 1*c*, *d*) though almost invariably plainly enough revealed by the tests to forbid grinding when the highest accuracy is of importance. Occasionally, as in 3*d*, fifteen minutes' grinding resulted in relatively appreciable oxidation of iron and absorption of moisture.

It is further very evident, as noticed by Mauzelius, that with different minerals very different degrees of oxidation result under like conditions, from a few per cent (1*e*) up to nearly 50 per cent (7) of the FeO after several hours' grinding, and it is not always the mineral that might *à priori* be regarded as most oxidisable that shows most oxidation. For instance, a magnesian siderite containing 46 per cent FeO, tested by myself, suffered oxidation of only about 3 per cent of its FeO when ground by itself for fifteen minutes, and but little over 5 per cent when ground for two hours with four times its weight of quartz powder. The nearly white colour of the original powder had, however, become decidedly brownish in both cases. It is undoubtedly true that, as shown by Mauzelius for mica, a soft or tough mineral undergoes greater oxidation if ground together with a hard one, like quartz as in the above instance, and with the mica tested by him, than by itself. This is due, of course, to the attrition effect of the harder mineral; hence, in different rocks the extent of oxidation of one and the same ferrous mineral may depend on the nature of its associates. Naturally the degree of water absorption will be less or greater for a like reason. Relatively slight oxidation of FeO after grinding in air (1, *c*, *d*) may sometimes be due to the hardness of the iron-bearing minerals or their toughness in absence of other hard ones, which would result in a far coarser powder than with some other rock or mineral ground for the same length of time, but in addition the relative stability of the chemical molecule is doubtless an important factor, perhaps the most important. Much remains to be done in accumulating experience with different minerals and their mixtures.

Naturally the idea of grinding in an inert atmosphere suggested itself, in order to prevent if possible the oxidation of FeO, if not the absorption of water. If the oxidation is due to local heating more than to surface exposure, this treatment should be effective in reducing or preventing it, and it is hoped to put it to the test for the purpose of settling the point more than with any hope of providing a practical means of preparing fine powders that shall be chemically unaltered. One test has already been made of grinding the siderite already mentioned in an atmosphere of carbon dioxide, which is suggestive, though not conclusive, because it was found impossible under the conditions to secure complete exclusion of air from the box in which the grinding was done. After two hours' grinding and subsequent thorough exposure to the air, the powder was darker than the unground material or than that ground under alcohol, but decidedly lighter in colour than that which had been ground for only hour in full contact with air. The inference is that local heating plays an important part in promoting oxidation. That an inert atmosphere would not be effective in preventing absorption of water was apparent, and was clearly shown by experiments 5*f* and 6, *d*, *e*, in which the grinding was done mechanically in an atmosphere dried by sulphuric acid, the mortar with its coarse powder having been placed in its housing together with vessels of sulphuric acid several hours before the grinding commenced, and a current of dry air passing through during the whole of the operation. The powder (6*e*) was then, after exposure to the air of the room for several hours, found to contain nearly as much water, loosely and firmly held, as when ground by the same machine for the same length of time in undried air. That the amounts were not fully as great is of little importance in view of the varying effectiveness of the different grinders, and even of the same grinder on different days.

Effect of Grinding under Liquids.

The results of grinding under different liquids are of interest, though not conclusive by reason of the limited number of tests that have been made. The results referred to in the foregoing paragraph rendered it unlikely that with the same degree of comminution there would be any material decrease in the amount of absorbed water, and experiments served only to confirm this view. They did show, however, that the effect was to materially reduce the oxidation of the iron, water being in this respect apparently somewhat less effective than absolute alcohol or carbon tetrachloride. A striking effect of the carbon tetrachloride may be mentioned. The mineral powder in contact with it becomes highly flocculated, and settles out at once when the grinding is interrupted, leaving a perfectly clear liquid above, in marked contrast to the lack of such effect with water and alcohol. As between the two named organic liquids, alcohol seems to be the more effective in preventing oxidation (2*b*, *c*, 5*b*, *c*, 10*b*, *c*) if we assume that the same degree of subdivision was reached with both in like periods of time, therefore the tests with carbon tetrachloride were not multiplied.

When alcohol was used, in most cases (1*b*, 3*b*, 10*b*, 12*b*) oxidation seems not to have taken place, even after two hours of continuous operation of the machine in the last two instances, at least not to a determinable extent. This again seems to be rather strong evidence that it is not increased surface exposure merely that gives rise to the marked oxidation of FeO when ground in air, but that local heating of the grains in contact with oxygen as they undergo crushing is the important factor. Were it not so, it would seem as if the iron should undergo oxidation after removal of the alcohol and exposure to the air, just as it then takes up water, but this it does not seem to do. In 2*b*, 5*b*, 9*b* for no evident reason the alcohol seems to have been less effective, taking always the FeO content of the crushed but unground bulk sample as the standard to go by. On the whole, this method of securing a fine sample does not altogether commend itself, since, while eminently satisfactory sometimes, the error affecting it is appreciable at other times. Nevertheless, it may give good service on occasion; hence the procedure to be followed in using it will be given. The coarse powder is covered in the mortar with absolute alcohol that leaves no residue whatsoever on evaporation. The amount used should not be more than enough to form a very liquid emulsion during the operation of grinding. One or two additions of alcohol may be needed if the grinding has to be of long duration. The grinding should be continued in any case till the mass becomes somewhat thick, then the mortar and pestle are left to dry by unaided evaporation of the excess of alcohol, a wide glass being supported above the mortar to keep out dust without too much impeding air circulation. When supposed to be thoroughly dry, the powder is transferred in its entirety to a watch-glass, and placed on the balance-pan, where it is to be kept till it is quite evident that no further loss in weight takes place. It is then transferred to a sample tube.

Application of the foregoing Results to Mineral Analysis.

It is plain from what has been said that nearly all mineral analyses that have been made in the past are affected by

more or less serious errors in respect to water and the oxides of iron, the error being greater the farther the comminution of the sample was carried. Very many analyses show more ferric iron in minerals that should apparently contain none, and small amounts of water for which there seems to be no place in any probable formula. These are discordances for which a simple and in most cases probable explanation now offers. A revision of much past analytical work, particularly that relating to iron-bearing minerals, is called for, but before it can be properly done, adequate provision must be made to avoid the old errors. In the paragraphs following will be set forth the procedures that seem best adapted to secure this end, in the light of present experience. It is perhaps advisable to add here, in order to avoid misconception, that the errors in past work are not, as a rule, to be assumed equal to those in the table that resulted from grinding for two hours or more, for few, if any, analysts have made it a rule to grind for such a length of time.

Treatment of Sample in First Stage of Analysis.—The mineral powder, prepared by careful crushing without grinding (see *ante*) serves as the starting-point. If it is possible to effect complete or nearly complete decomposition within thirty minutes by dilute hydrofluoric acid with powder so coarse as just to pass a sieve of 80 meshes to the linear inch (about 0·20 mm. diameter of apertures), a powder of this degree of fineness should be used in preference to one finer. In general, such a powder can be used without grinding for all determinations that involve fusion with an alkali carbonate, but not always. If finer grinding, either in air or under alcohol (see above) is imperative, it will be necessary with the ferrous iron portion, when there is enough of this constituent to make an appreciable difference, to make water determinations on the finely-ground as well as on the coarse powder, in order to be able to reduce the results obtained for the ferrous oxide to the weight represented by the coarse powder. For other constituents, as the alkalis, this course will not generally have to be followed; it will be sufficient to take of the coarse sample a weight called for by the particular determination, and to grind this in a large mortar with extreme care to avoid loss by dusting or flying of grains, and then to cleanse the mortar by grinding in it a little of the flux that is to be subsequently employed, or of some harmless material other than a flux, the rinsings being added, of course, to the main portion of the fine powder.

For the ferrous iron determination two courses are open:—(*a*) To grind under alcohol until a degree of fineness is reached that will surely admit of complete decomposition by boiling dilute hydrofluoric acid within fifteen or twenty minutes; (*b*) to treat the coarse powder directly with hydrofluoric acid. If a single treatment by (*b*) does not effect complete decomposition, and this will be very often the case, the procedure that has thus far commended itself is the following:—Having titrated (according to the directions in *Bull. U.S. Geol. Survey*, cccv., 142) with permanganate to the first more or less transient pink blush throughout the whole body of liquid, let settle, decant, and wash once by decantation with water, rinse the residue into a small agate mortar, decant most of the water after again allowing to settle, grind the residue for a short time under what water remains, transfer it by a stream of hot water to the large crucible from which it was taken, and repeat the treatment by hydrofluoric and sulphuric acids used in smaller amounts than the first time. The final titration may be made in the crucible itself if this is of at least 80 cc. capacity and the bulk of liquid in it is small, after quickly diluting with cold water till the crucible is nearly full.

This manner of treatment has afforded thus far satisfactory results, and is in most cases to be preferred to that given as (*a*). Not only are the double determinations of water avoided, but also the error that often arises from oxidation by long grinding under alcohol. The very short grinding under water of the small fraction of the original iron-bearing mineral or mineral that remains after the first treatment with hydrofluoric acid cannot conceivably lead to any detectable error.

In conclusion, a few remarks are in place regarding the comparative merits of the Pratt and Cooke methods of decomposing minerals for the ferrous iron determination. In *Bull.*, cccv., 138, it was said that indications seemed to slightly favour the former method, and this expression is strengthened by the comparative data contained in the table. Although in some instances (2, *b*, *e*, 5, *a*, *b*, *d*) the two methods appear to be of equal merit, in others (2, *a*, *f*) they differ materially in favour of that of Pratt, and in the rest there is evident a general tendency toward lower results by that of Cooke.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

Part III.—Analysis of the Aluminium and Iron Groups, including Beryllium, Uranium, Vanadium, Titanium, Zirconium, and Thallium.

By Arthur A. Noyes, William C. Bray, and Ellwood B. Spear.

(Continued from p. 210).

Confirmatory Experiments and References (continued).

Solubility of Aluminium Hydroxide in Excess of Ammonia.—To a solution containing 10 mgrms. Al as nitrate and 4 cc. HCl (1·12) in 100 cc. were added in the cold 17 cc. NH_4OH (0·96); *i.e.*, an excess of about 10 cc., and the mixture was shaken; the precipitate of $Al(OH)_3$ was filtered off after about five minutes, and the filtrate was boiled for several minutes to expel the excess of NH_4OH; a precipitate of $Al(OH)_3$ separated which was estimated to contain about 3 mgrms. Al. The experiment was repeated, except that an excess of only 2 or 3 cc. NH_4OH (0·96) was used; the filtrate on boiling became turbid, but the amount of aluminium that separated did not exceed 0·5 mgrm.

P. 51, N. 3 *and* 4: *Solubility of* $Mg(OH)_2$ *in* NH_4Cl *Solutions.*—The solubility of $Mg(OH)_2$ in water is about 2×10^{-3} mols. per litre. For evidence that the non-precipitation of $Mg(OH)_2$ in moderately dilute solutions containing an ammonium salt depends solely on the driving back of the dissociation of NH_4OH, see Lovén (*Zeit. Anorg. Chem.*, 1896, xi., 404), Treadwell (*Zeit. Anorg. Chem.*, 1904, xxxvii., 326), Herz and Muhs (*Zeit. Anorg. Chem.*, 1904, xxxviii., 138).

The following experiments show that enough ammonium salt is produced by the neutralisation of the 4 cc. HCl (1·12) originally present to prevent the precipitation of $Mg(OH)_2$ even when 500 mgrms. are present. A solution containing 500 mgrms. as $MgCl_2$ and 4 cc. HCl (1·12) in 100 cc. was neutralised with NH_4OH (0·96, 6 cc.), and more NH_4OH was added. No precipitate formed when 40 cc. in all had been added, but the addition of 10 cc. more gave a precipitate. The experiment was repeated with an initial volume of 30 cc. instead of 100 cc.; 30 cc. NH_4OH produced no precipitate, even after several minutes, but 40 cc. did. The experiment was repeated with 2 cc. HCl and a volume of 30 cc.; 15 cc. NH_4OH (0·96) produced no precipitate, but 20 cc. did.

Complex Zinc Ammonia Cation.—$Zn(NH_3)_4{+}{+}$. See Herz (*Zeit. Anorg. Chem.*, 1900, xxv., 225), Gaus (*Zeit. Anorg. Chem.*, 1900, xxv., 236), Euler (*Ber.*, 1903, xxxvi., 3400), Bonsdorff (*Zeit. Anorg. Chem.*, 1904, xli., 132).

Complex Nickel Ammonia Cation.—$Ni(NH_3)_4{+}{+}$.—See Dawson and McCrae (*Journ. Chem. Soc.*, 1900, lxxvii., 1239), Konowaloff (*Chem. Centralblatt*, 1900, i., 646), and especially Bonsdorff (*Zeit. Anorg. Chem.*, 1904, xli., 132).

Behaviour of Chromium towards Ammonium Hydroxide.—To a solution containing in 30 cc. 3 or 4 cc. HCl (1·12) and 250 mgrms. Cr as $CrCl_3$ freshly prepared by boiling K_2CrO_4 with concentrated HCl, was added NH_4OH (0·96)

* From the *Journal of the American Chemical Society*, xxx., No. 4.

until after shaking the odour was distinct. The mixture was divided into two parts; one part was filtered at once; to the other part 10 cc. NH_4OH (0·96) were added, and it was immediately filtered; the former had only a very faint pink colour, and on boiling not more than 0·5 mgrm. Cr precipitated as $Cr(OH)_3$; the latter was distinctly pink, and on boiling 2 to 3 mgrms. Cr precipitated as $Cr(OH)_3$. The experiment was repeated, except that an excess of 2 cc. NH_4OH was added after the odour of NH_4OH was distinct, and that in addition 5 grms. solid NH_4Cl were added to one-half of the mixture, and both portions were filtered at once; each filtrate was faintly pink, and on boiling that containing no excess of NH_4Cl there resulted a precipitate of $Cr(OH)_3$ estimated to contain 0·5 mgrm. Cr, while on boiling the other filtrate no precipitate separated, and the solution remained pink. The last experiment was repeated, except that the mixtures were allowed to stand twenty hours before filtering; the filtrate containing no excess of NH_4Cl was faintly pink, but did not contain more than 1 mgrm. Cr. The second filtrate was highly coloured, and on boiling 5 to 10 mgrms. Cr precipitated as $Cr(OH)_3$, but the precipitation was not quite complete. These results show that both NH_4OH and NH_4Cl increase the amount of the pink chromium compound formed, and therefore prove that this compound is a complex ammonia salt and not a chromite. For the proof that these solutions are unstable, even at room temperatures, see Herz (*Zeit. Anorg. Chem.*, 1902, xxxi., 357).

P. 51, N. 6: *Precipitation of Phosphates of Calcium, Barium, Magnesium, and Manganese by Ammonium Hydroxide.*—To solutions containing 2 mgrms. PO_4 as Na_2HPO_4 and 4 cc. HCl (1·12) in 30 cc., were added in separate experiments varying amounts of manganese, barium, and magnesium as chloride, and of calcium as nitrate. The solutions were then neutralised with NH_4OH (0·96) and about 2 cc. excess added, making 8 cc. in all, after which the mixtures were shaken and allowed to stand; 2 and 3 mgrms. Mn gave small precipitates after a minute or two, but 1 mgrm. gave no precipitate; 10 and 20 mgrms. Ca gave large precipitates at once, but 5 mgrms. gave no precipitate even in half-an-hour. 100 mgrms. Ba gave a large precipitate at once, and 50 mgrms. gave no precipitate in half-an-hour. One, 3, and 5 mgrms. Mg gave precipitates on shaking. The experiment was repeated with 10 mgrms. PO_4 and varying amounts of barium. Sixty mgrms. Ba gave a large precipitate at once, but 40 mgrms. gave no precipitate, or only a very small one, in half-an-hour. The experiment was repeated with 2 mgrms. Ca and varying amounts of phosphate; with 30 mgrms. PO_4 a precipitate was obtained at once, with 20 mgrms. after several minutes, and with 10 mgrms. no precipitate appeared in half-an-hour. On repeating the experiment with 1 mgrm. Ca a precipitate was obtained with 30 mgrms. PO_4 after a few minutes, but not with 20 mgrms.

P. 51, N. 7: *Solubility of the Borates of the Alkaline Earth Elements.*—500 mgrms. BO_2 as H_3BO_3 and 20 mgrms. Ca as chloride were dissolved in 4 cc. HCl (1·12) and 36 cc. water, and NH_4OH (0·96) were added until the mixture after shaking just smelled of it; no precipitate separated. The experiment was repeated with 200 mgrms. Ba as chloride; no precipitate separated. The experiment was repeated with 300 mgrms. Ba; a small precipitate formed, but it dissolved on adding a little concentrated NH_4Cl solution. The experiment was repeated with 500 mgrms. Mg as chloride; no precipitate resulted.

P. 51, N. 9: *Action of Ammonium Hydroxide on Uranyl Solutions.*—See Kern (*Journ. Am. Chem. Soc.*, 1901. xxiii., 701). One and 3 mgrms. U as $UO_2(NO_3)_2$ were dissolved in separate experiments in 20 cc. water, and a few drops NH_4OH (0·96) were added; no precipitate separated on standing, nor on boiling. A small quantity of solid $NaNO_3$ was added to the cold solutions; pale yellow precipitates separated at once, showing that the uranium had been present in the colloidal form. The experiment with 1 mgrm. U was repeated, except that a little NH_4Cl instead of $NaNO_3$ was added; a yellow precipitate separated.

P. 51, N. 9: *Uranyl Salts and Diuranates.*—See Dittrick (*Zeit. Phys. Chem.*, 1899, xxix., 449), Kern (*Journ. Am. Chem. Soc.*, 1901, xxiii., 686).

P. 51, N. 10: *Completeness of Precipitation of the Sulphides of the Iron Group.*—In each of the following experiments the quantity of the element given below was dissolved in 4 cc HCl (1·12) and 96 cc. water; to this solution in the cold were added 9 to 10 cc. NH_4OH (0·96), and then $(NH_4)_2S$ solution drop by drop until an excess was present. The mixtures were well shaken, and then filtered, generally through a double filter. The results were as follows:—With 5 mgrms. Mn as $MnCl_2$, a light coloured precipitate was formed at once; the filtrate was clear after two filtrations. With 0·5 mgrm. Mn as $MnCl_2$, the result was the same. With 0·25 mgrm. Mn as $MnCl_2$, the solution became turbid in two or three minutes. The filtrate in the first two experiments was evaporated almost to dryness; HNO_3 (1·42) was added; the mixture was evaporated to 5 cc., $KClO_3$ was added, and the mixture boiled; no precipitate of MnO_2 separated, showing that the precipitation of MnS had been complete.

With 1 mgrm. Zn as $Zn(NO_3)_2$, the mixture became turbid at once, and was readily filtered. With 0·5 mgrm. Zn, the result was the same. The filtrate in the first experiment was evaporated to 5 cc., made alkaline with NH_4OH and $(NH_4)_2S$ was added; only an insignificant turbidity appeared, showing that the precipitation of ZnS was practically complete.

With 0·5 mgrm. Co as $Co(NO_3)_2$, a black precipitate formed at once, which was readily filtered off. With 0·1 mgrm. Co, the solution became dark coloured at once.

With 0·2 mgrm. Fe as $FeSO_4$, the solution became dark coloured at once, and the precipitate was easily filtered off. With 0·5 mgrm. Fe as $FeSO_4$, the result was the same. The filtrate was evaporated to 5 cc.; NH_4OH and $(NH_4)_2S$ were added; the solution remained colourless.

With 0·3, 0·5, and 1·0 mgrm. Tl as Tl_2SO_4, the solutions became dark brown at once, and yielded clear filtrates, which when evaporated almost to dryness and tested for thallium by *P. 65*d*, gave no precipitate of Tll.

With 0·5 and 1·0 mgrm. U as $UO_2(NO_3)_2$, distinct flocculent precipitates could be seen after the addition of $(NH_4)_2S$; the filtrates were clear, and the precipitate, when collected on the filter, was yellow in colour. With 0·3 mgrm. U as $UO_2(NO_3)_2$, a slight coloration was seen on the filter, but the precipitate could scarcely be seen before filtration. With 5 mgrms. U as $UO_2(NO_3)_2$, the result was the same. The filtrate was evaporated almost to dryness, and was tested for uranium by *P. 58*d with* $K_4Fe(CN)_6$; no brown coloration was observed. The last experiment was repeated, except that the filtrate was evaporated to 5 cc. and NH_4OH and $(NH_4)_2S$ added; no precipitate formed, showing that the precipitation as sulphide in 100 cc. had been complete.

Nature of the Uranium Precipitate produced by Ammonium Sulphide.—Twenty mgrms. U as $UO_2(NO_3)_2$ were dissolved in 4 cc. HCl (1·12) and 96 cc. water, about 9 cc. NH_4OH were added, and $(NH_4)_2S$ drop by drop in the cold, until a distinct excess was present. The precipitate was flocculent and had a bright yellow colour. After two hours it was light brown, and in six hours dark brown in colour. The experiment was repeated, except that a much larger excess of $(NH_4)_2S$ was added; the precipitate darkened more rapidly. It is evident that $(NH_4)_2U_2O_7$ is first precipitated contrary to the statements in some text-books, and that the conversion of this substance into UO_2S takes place slowly.

The Existence of Ferric Sulphide (Fe_2S_3).—That Fe_2S_3 is formed when $(NH_4)_2S$ is added to suspension of $Fe(OH)_3$ in dilute NH_4OH has been proved by Stokes (*Journ. Am. Chem. Soc.*, 1907), xxix., 304. When, however, an acid solution is first saturated with H_2S and then made alkaline with NH_4OH, FeS and not Fe_2S_3 is formed.

P. 51, N. 11: *Dark Colour of Sulphide Precipitate as Evidence of Iron, Nickel, or Cobalt.*—To solutions containing 500 mgrms. Zn. as nitrate, 4 cc. HCl (1·12) and

30 cc. H_2O were added in separate experiments 1 and 2 mgrms. Ni as nitrate, 1 and 2 mgrms. Co as nitrate, 1 mgrm. Fe as $FeSO_4$, and 1 mgrm. Fe as $FeCl_3$. The mixtures were neutralised with NH_4OH (0·96) and 3 or 4 cc. excess added, making 10 cc. in all. A large white precipitate of $Zn(OH)_2$ remained which was estimated to contain at least 100 mgrms. Zn. Colourless $(NH_4)_2S$ was then added as described in P. 51, and the colour of the precipitate was compared with that obtained with 500 mgrms. pure zinc; the precipitates containing 1 mgrm. Fe were nearly black with a greenish tinge; those containing 2 mgrms. Ni and Co were much darker than the pure ZnS, but those with 1 mgrm. Ni and Co were only very slightly darker. The experiment was repeated except that 4 cc. more $(NH_4)OH$ were added in order to dissolve the $Zn(OH)_2$; the results were substantially the same. A solution containing 500 mgrms. Ca and 1 mgrm. Ni as nitrates, 1 grm. PO_4 as $(NH_4)_2HPO_4$, 4 cc. HCl (1·12) and 35 cc. H_2O was made alkaline with NH_4OH (0·96) and 1 cc. colourless $(NH_4)_2S$ was added: the large white precipitate of phosphate became dark coloured as soon as the sulphide was added.

P. 51, N. 12: *Behaviour of Nickel towards Ammonium Monosulphide and Polysulphide.*—See Lecrenier (*Chem. Zeit.*, 1889, xiii., 431, 449), Anthony and Magri (*Gazz. Chim. Ital.*, 1901, xxxi., II., 265). By boiling the brown solution in the absence of air the last-named authors have prepared NiS_4. The composition of the brown solution is unknown; it may be ammonium sulpho-nickelate, $(NH_4)_2NiS_{2+x}$, or colloidal nickel persulphide.

That in absence of air nickel is completely precipitated as NiS and the filtrate is colourless, thus proving that the brown solution is due to the presence of polysulphide, has been shown by Lecrenier (*Chem. Zeit.*, 1889, xiii., 431, 449) and Villiers (*Comptes Rendus*, 1894, cxix., 1263). The presence of NH_4OH tends to prevent the precipitation of NiS, and it is possible to obtain colourless (or bluish) solutions, containing a small excess both of $(NH_4)_2S$ and of nickel. In the presence of air we have found it almost impossible, when working with large amounts of a pure nickel salt, to prevent some nickel from passing into the filtrate, giving a brown solution. The amount of nickel in the filtrate increased with the excess of ammonium sulphide used and with the length of exposure to the air.

Some of the brown solutions obtained in these experiments were boiled in small flasks for three to ten minutes, and then filtered; in each case the filtrate was colourless after a single filtration.

Precipitation of Nickel in Ammoniacal Solution by Hydrogen Sulphide.—In several experiments 500 mgrms. Ni as $Ni(NO_3)_2$ were dissolved in 30 cc. water and 4 cc. HCl (1·12); NH_4OH (0·96) was added until the odour could be detected after shaking and then 3 cc. more, and H_2S was led into the mixture for fifteen minutes; the precipitates were always granular, and the filtrates clear and colourless; when the precipitates were washed with water containing either H_2S or a little $(NH_4)_2S$ the wash water was invariably clear.

(To be continued).

NOTICES OF BOOKS.

Forty-fourth Annual Report on Alkali, &c., Works. By the CHIEF INSPECTOR. London: Eyre and Spottiswoode, Ltd. 1908.

THIS annual report contains a condensed account of the important work published by Dr. Raschig during 1907 on the nature of the reactions which occur in the lead chamber process, and also a full report is given of the investigations of Mr. Linder on the use of "Dolly Cream" dye as a selective indicator for the detection of certain acids in gaseous mixtures. By means of these investigations the exact uses and limitations of this indicator have been defined. The work of Prof. Percy Frankland and Mr. H. Silvester on the bacterial treatment of spent ammoniacal liquors is also summarised.

Das Erdöl, seine Verarbeitung und Verwendung. ("Petroleum, its Preparation and Applications"). By Dr. RICHARD KISSLING. Halle-a-S.: Wilhelm Knapp. 1908.

THIS is one of the best of the smaller books on petroleum which has come under our notice of late years. It is intended principally for those chemists who want to read through an introductory survey before proceeding to the study of larger treatises, and for this purpose they will find it excellent. The author has endeavoured to present a many-sided view of the subject, at the same time carefully avoiding any excess of detail, and throwing the information into the most concise form possible. The matter is so well chosen that the reader will have nothing to unlearn when he passes on to more detailed work, and, moreover, all that he has learnt will form a ground-work upon which further knowledge can very readily be based. The distribution and supply, the chemical nature, preparation, and uses of petroleum are all discussed as far as possible in general terms, and typical methods and processes are always chosen for consideration. The bearings of theory upon practice receive careful attention, and all points of special practical signification are given the prominence which their importance demands. A thoroughly systematic and well-arranged treatment of each division of the subject accounts for the large amount of matter contained in a small space, and every branch of the petroleum industry is discussed in short accounts in which no essential points are overlooked.

Laboratoriumsbuch für die Industrie der Riechstoffe. ("Laboratory Book for the Perfumery Industry"). By Dr. OSKAR SIMON. Halle-a-S.: Wilhelm Knapp. 1908.

THE first part of this book contains a description of those methods of analysis which may most conveniently be applied to identify, or test the purity of, the most important perfumes. Both physical and chemical methods of investigation are included, and the experimental directions are full enough to enable anyone who has had some general training in practical chemistry to perform the tests without further assistance and without much difficulty. The separation of hydrocarbons from ethereal oils and their identification is a subject which is rather scantily treated, some important hydrocarbons, as, for instance, thuyene and styrol, being omitted. In the second part of the book short accounts are given of the most common perfumes; their origin is discussed, and their physical constants are enumerated; some notes are also added on modifications of the general methods of analyses which may conveniently be used in special cases.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 13, September 28, 1908.

Relative Stability of the Cyclic Poly-carbon Groups. — Louis Henry. — Dehydrating agents act on dimethylisopropylcarbinol, $(H_3C)_2=C-CH=(CH_3)_2$ (OH), giving the two unsaturated isomeric hydrocarbons—tetra-

methylethylene, $\begin{matrix}CH_3\\CH_3\end{matrix}\!>\!C=C\!<\!\begin{matrix}CH_3\\CH_3\end{matrix}$, and methylisopropylethylene, $\begin{matrix}CH_3\\CH_3\end{matrix}\!>\!CH-\underset{\displaystyle CH_3}{\overset{|}{C}}=CH_2$, in the proportion of three parts of the former to one of the latter. The investigation of the corresponding tertiary albohol containing the cyclic group $H_2C\!<\!\begin{matrix}CH_2\\CH_2\end{matrix}\!>\!CH,-$

$$H_2C\!<\!\begin{matrix}CH_2\\CH_2\end{matrix}\!>\!CH-\underset{\displaystyle OH}{\overset{|}{C}}H-C\!<\!\begin{matrix}CH_3\\CH_3\end{matrix}$$

shows that the group $H_2C\!<\!\begin{matrix}CH_2\\CH_2\end{matrix}\!>\!CH$ is relatively stable, and the elimination of H—OH occurs at the expense of the system $-\underset{\displaystyle OH}{\overset{|}{C}}\!<\!\begin{matrix}CH_3\\CH_3\end{matrix}$.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xli., No. 13, 1908.

Preparation of Hydrazoic Acid.—Johannes Thiele.—The conversion of hydrazine into hydrazoic acid by nitrous acid, by oxidation either alone or in presence of hydroxylamine, or by the action of nitrogen chloride, gives only small yields. But if ethyl nitrite is allowed to act on hydrazine in presence of alkali, good yields of sodium nitride are obtained, especially if excess of nitrite is used. Ethyl nitrite is not saponified by alkali in the cold, and the free hydrazoic acid can be obtained in aqueous solution by allowing hydrazine sulphate to react with ethyl nitrite in presence of caustic soda. Sodium nitride can also be obtained by heating hydrazine hydrate with sodium nitrite in aqueous solution. The reaction takes place slowly, and is incomplete; it is accelerated by the presence of some hydrazine sulphate, or by large quantities of sodium hydroxide.

Reduction of Nitro-compounds with Zinc Dust and Acetic Acid.—Gustav Heller.—When different orthonitro compounds are subjected to the same reduction process with zinc dust and acetic acid, the results obtained vary apparently irregularly, but the residue in the ortho-position appears to facilitate the reaction. Thus, orthonitromandelic acid nitrile is very readily reduced at o°, while under the same conditions nitrobenzene is almost unchanged. Phenylhydroxylamine is formed, for the aqueous solution gives phenylazohydroxylanilide with diazobenzene chloride. *o*-Nitrobenzaldehyde gives *o*-aminobenzaldehyde and anthranil. From the products of the reduction of *o*-nitrobenzoic acid, benzidine dicarboxylic acid and azoxybenzoic acid can be isolated.

Molecular Weight and Hydrate of Hypophosphoric Acid.—Arthur Rosenheim and Max Pritze.—Hypophosphoric acid esters (ethyl and methyl), and pyrophosphoric acid ester were prepared by the action of alkyl iodide on silver hypo- or pyrophosphate, and their molecular weights were determined by the boiling-point method. The pyrophosphoric acid esters gave values which agreed very closely with the molecular weights of the substances of formulæ $(CH_3)_4P_2O_7$, $(C_2H_5)_4P_2O_7$, while with the same solvents the corresponding hypophosphoric esters give numbers which point to the simple formula H_2PO_3 for the acid. When a solution of free hypophosphoric acid is allowed to crystallise over sulphuric acid *in vacuo*, tabular transparent crystals are formed. They melt at 62°, and the analysis of them shows that they are the monohydrate, $H_2PO_3.H_2O$. This monohydrate is stable between o° and 60°.

New Synthesis of Aromatic Carboxylic Acids from Hydrocarbons.—Paul Schorigin.—When carbon dioxide acts on a mixture of sodium and mercury alkyl in benzene solution a fatty acid and benzoic acid are formed. Apparently a sodium alkyl is formed as an intermediate product, the reaction occurring in phases as follows:—

$$\text{I.}\quad Hg(C_2H_5)_2 + 2Na = Hg + 2NaC_2H_5.$$
$$\text{II.}\quad C_6H_6 + NaC_2H_5 = NaC_6H_5 + C_2H_6.$$
$$\text{III.}\quad C_6H_5Na + CO_2 = C_6H_5.COONa.$$

Mercury diethyl can be replaced by zinc diethyl in this reaction. When this reaction was extended to other aromatic hydrocarbons it might be expected that the sodium and carbon dioxide would enter the benzene nucleus, but, on the contrary, it is found that when mercury diethyl is used the acids with the carboxyl in the side-chain result. Apparently this new method of synthesing carboxylic acid can only be applied to the benzene derivatives. The reaction is caused by the great lability of sodium alkyls.

Triphenylstibine Sulphide.—Ludwig Kaufmann.—The cautious introduction of sulphuretted hydrogen into an alcoholic ammoniacal solution of triphenyl stibine leads to the separation of white crystalline needles of triphenylstibine sulphide; further introduction of sulphuretted hydrogen turns the liquid an intense yellowish red, and from this liquid triphenylstibine can be isolated. In all probability the hydroxide, $(C_6H_5)_3Sb(OH)_2$, is formed as an intermediate product, and then decomposed quantitatively according to the equation $(C_6H_5)_3Sb(OH)_2 + H_2S = (C_6H_5)_3SbS + 2H_2O$. When the sulphide is subjected to the action of sulphuretted hydrogen it dissolves, and a yellow coloration is produced; this is due to the antimony compound formed from the sulphide, and dissolved in alcohol. It is possible that on solution a sulpho-salt or a derivative of tetravalent sulphur is formed, $(C_6H_5)_3Sb\!<\!\begin{matrix}S.NH_4\\SH\end{matrix}$ or $(C_6H_5)_3Sb:S:S\!<\!\begin{matrix}NH_4\\H\end{matrix}$. Whatever the nature of the intermediate substance, it is very labile and readily undergoes decomposition. If a solution of the sulphide in alcoholic ammonium sulphhide is prepared in such a way that an excess of ammonium sulphide is avoided, and the solution is filtered and allowed to stand, the colourless liquid gives only triphenylstibine when it evaporates in a vacuum desiccator.

Action of Magnesium Organic Compounds on Arsenious Acid.—Franz Sachs and Hans Kantorowicz.—Although arsenious acid is praetically insoluble in ether, when it is finely powdered and introduced into Grignard's solution a reaction occurs; heat is evolved, and the acid disappears. The reaction is complicated, and, according to the organo-metallic components, different final products are obtained. Thus, in the cold, diphenylarsenic oxide is obtained when phenyl magnesium bromide is used, while the product is triphenylarsine when the temperature is raised, $C_6H_4(CH_3)$. MgBr gives only tritoluylarsine, but benzyl magnesium chloride gives a compound which is probably $(C_6H_5.CH_2)_2As.OH + H_2O$.

MEETINGS FOR THE WEEK.

MONDAY, Nov. 2nd.—Royal Institution, 5. General Monthly Meeting.
Society of Chemical Industry, 8. "Chemical Industry in Relation to Agriculture," by A. Frank.

THURSDAY, 5th.—Chemical, 8.30. "Direct Union of Carbon and Hydrogen," by W. A. Bone and H. F. Coward. "Relation between Absorption Spectra and Chemical Constitution—Part XI., Some Aromatic Hydrocarbons," by E. C. C. Baly and W. B Tuck. "Organic Derivatives of Silicon—Part VII., Synthesis of *dl*-Sulphobenzylethylisobutylsilicyl Oxide," by B. D. W. Luff and F. S. Kipping. "Chlorine Derivatives of Pyridine—Part IX., Preparation and Orientation of the Dichloropyridine M. p. 66–70°; Part X., Orientation of the Trichloropyridine M. p. 49–50°" and "Chlorination of the Methyl Derivatives of Pyridine—2-Methyl Pyridine (Part II.)," by W. J. Sell. "The Triazo-group—Part V., Resolution of *a*-Triazopropionic Acid; Part VI., Triazoethyl Alcohol and Triazoacetaldehyde," by M. O. Forster and H. E. Fierz.

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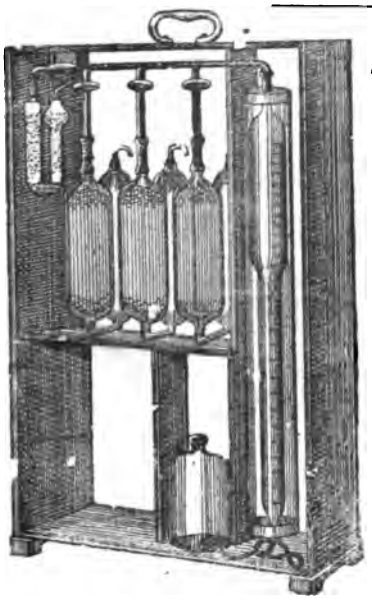

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By C. AUER VON WELSBACH.

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ON page 11 of my article which appeared last year, "On the Elements of the Ytterbium Group," Part I., I mentioned that the last member of all ytterbium fractions from the purified final mother-liquors of the principal series was a fraction the spark spectrum of which showed striking differences from the normal ytterbium spectrum. For example, the 4-line group in the green ($\lambda = 5353{\cdot}0$, $5347{\cdot}4$, $5345{\cdot}9$, $5335{\cdot}2$), which is so characteristic of ytterbium, was much weakened; other lines (mostly fainter) were altogether missing.

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As the spark spectrum of the end fraction consisted exclusively of ytterbium lines, from the changes of intensity which the lines had undergone, it must be concluded that one constituent of ytterbium had accumulated in the more soluble fractions, so that ytterbium cannot be an elementary substance.

Other observations also pointed to the same conclusion, long before the discovery of this spectral phenomenon. For instance, it was very remarkable that the separation process for erbium and thulium on the one hand, and ytterbium on the other, was effected more slowly the longer it was continued. While at first after a few series colourless ytterbium salts giving no absorption spectrum could be obtained, this became gradually more difficult, although the ytterbium fractions put aside as pure remained almost unchanged in amount. If it was proved that on fractionating the double ammonium oxalates one constituent of the ytterbium—for the present I shall call it Yb I.—had accumulated in the more soluble fractions, naturally after a correspondingly lengthy fractionation this constituent must have decreased in the difficultly soluble double salts, and consequently another constituent of ytterbium must have increased.

But these difficultly soluble double salts are those fractions containing little erbium which occur between the colourless ytterbium fractions and those richer in erbium. As solid salts all these fractions show, besides the Tu bands, that remarkable spectrum which is quite different from the absorption spectrum of pure erbium salts, and which I called the spectrum Er γ in the article mentioned above.

I first set to work on these fractions. I added to them later all those erbium fractions in which the holmium lines could no longer be detected.

The fractionation was performed as before. As after fifty series it was continually becoming more difficult to get quite colourless fractions, and the number of the fractions of the series was continually increasing, I removed the very faintly pink-tinged fractions which gave the above-mentioned Er γ spectrum, and put them together in several collective fractions.

The mother-liquors of these series were evaporated, and gave small quantities of perfectly colourless fractions which were added to the yttterbium fractions previously obtained.

After the seventy-fifth series I was putting aside salts which were more strongly pink coloured than those removed before, and which all showed to a slight extent the characteristic bands of the erbium spectrum, besides the spectrum Er γ.

* *Monatshefte für Chemie*, February, 1908.

The mother-liquors of these series were no longer put with the others. They finally gave, after repeated evaporation, a fraction which hardly showed the erbium spectrum, Mother-liquor I.

Putting aside the middle pure erbium fractions, to which I shall not allude further in this article, I allowed the crystallisation process to go on to the end. These last series gave, finally, a final Mother-liquor II. and a large quantity of faintly pink-coloured salts which all gave the spectrum Er γ.

Mother-liquor I., when precipitated with nitric acid, gave an almost white oxalate. It contains chiefly ytterbium.

Mother-liquor II. gave a very pale pink oxalate.

In all these fractions a small amount of elements allied to tin was present. I shall deal in a special article with these interesting substances which occur in all minerals containing the elements of the Yb group.

In the spark spectrum of the pure oxide prepared from the oxalate of Mother-liquor II. there appeared very clearly the countepart of the spectrum described at the beginning of this article. All ytterbium lines which appeared paler in that spectrum were brighter in this spectrum, especially the 4-line group in the green. The spectrum of this second ytterbium constituent—it may provisionally be called Yb II.—appeared still more clearly in the faintly pink-coloured fractions which followed Mother-liquor II. In the spectra of these preparations the lines which appear so brilliant in the spectrum, described at the beginning of this article, can now hardly be seen.

As these spectra contained only the lines of Yb II., besides some erbium lines, it may be concluded that the substance causing the pink coloration of these fractions either possesses no sensitive spark spectrum, or else is present in traces only. The first assumption suggests erbium. The spectrum of this substance is not specially bright in the visible part, although it contains many lines. Since, on the other hand, the substance giving the spectrum Er γ cannot be appreciably increased in the fractions, it may be assumed that this special spectrum, which has so often given rise to mistakes, is a variation of the erbium spectrum.

When I dissolved by boiling a mixture of ten parts of the purest ytterbium-ammonium oxalate and one part of the purest erbium ammonium oxalate in a solution containing as little ammonium oxalate as possible, and then allowed it to crystallise quickly, a double salt was formed which did not differ appreciably in colour nor in absorption spectrum from the fractions in question.

For completeness sake it must be mentioned that the spectra of all fractions containing a little erbium also showed clearly the absorption bands ascribed to thulium.

By the experiments just described it was shown that all faintly pink-coloured fractions contained about ten times more Yb II. than erbium. The spectrum Er γ was henceforward employed as a convenient indicator of the amount of ytterbium in the fractions.

Owing to the high percentage of Yb II. in the faintly pink-coloured fractions, it occurred to me to attempt the separation of this constituent of ytterbium by the basic nitrate method—a method which has proved effectual for the preparation of ytterbium.

After a preliminary experiment with a little pure salt had demonstrated the applicability of this method, I applied it to these fractions.

As I have already described the exact process in my earlier articles, I shall here report shortly on the results only. At first the separation of the constituents occurred in the normal way, but as soon as the amount of erbium had sunk below about $\frac{1}{2}$ per cent the separation became more difficult, so that it was only possible to get perfectly colourless erbium-free products by repeated fractionations. This somewhat negative result shows that the separation by means of the double ammonium oxalates is not only the more convenient but also the far better method.

I now arranged the fractions containing the ytterbium

elements into a new series. This was rather difficult, because the preparations came from different stages in the separation process, and contained very different amounts. I first got the above-mentioned faintly pink-coloured collective fractions into a series which consisted of ten fractions. At the head of these I placed those fractions which I had prepared from the mother-liquor of the basic salts. At the end came some other preparations of the last series containing much more erbium, so that this new series consisted of about sixteen members. The separation process then began again. As it went on I interpolated in the series the other ytterbium fractions of the principal series. Some of them gave no trace of absorption bands in the spectrum, but others showed them just plainly enough to be recognised. I shall henceforward denote the elements giving these absorption bands collectively as "erbium." Most fractions contained a large excess of ammonium oxalate, much more than was necessary to dissolve the salt on warming.

This excess must first be removed, because it would make the separation process from a hot solution difficult. For this purpose I prepared a solution of the mixture of salts saturated at about 90°, and then let it crystallise in a thermostat at 60°. After about twelve hours the solution was rapidly poured off from the ammonium oxalate separated, which had formed a crystalline mass adhering rather closely to the dish, into a previously warmed dish. By repeated crystallisations at about 30° C., it was easily possible to separate the ammonium oxalate nearly pure. In the liquor poured off from the crystals the other fractions were dissolved, treated in the way described above, and so on, until all were in solution in different portions. I kept these solutions more concentrated the more impure they were.

The separation of the first fractions was always performed at about 50° C. As soon as the amount of erbium had fallen below ½ per cent I let the liquors crystallise at room temperature. Corresponding fractions were put together and united to form a new series.

The mother-liquors arising from this series were repeatedly concentrated fractionally. The mother-liquors finally obtained, in which, besides rare earths, other impurities had accumulated, arising partly from the water used, were carefully precipitated with nitric acid. The precipitate consisting of earth oxalates and acid ammonium oxalate was filtered off, washed with cold water containing some oxalic acid, then suspended in water, and dissolved by heating, with the addition of a little ammonia. By putting together the corresponding fractions a new series was finally formed.

As the separation process was continued gradually the final members of the other series could be added to the new fractions, until, putting aside the faintly pink-coloured first members, all the fractions could be united in a single series.

This series contained nearly half a kilogrm. of ytterbium oxide.

It would be very tedious and wearisome if I were to give a detailed description of the subsequent separation process. I shall therefore only shortly allude to the most important steps in the process.

After the tenth series had been formed I put aside the crystal aggregate of the first fractions, and repeated this in almost every new series. Finally, I had forty crystalline fractions and a larger quantity of final mother-liquors as collective fractions. The first ten fractions amounted to about two-thirds of the total quantity. The first showed a still recognisable pink coloration, but all the rest were perfectly white, and gave no visible absorption spectrum, even with a deep layer. The spark spectrum of the first fractions contained only the lines of Yb II. In the next fractions the most intense lines of Yb I. could be detected. Towards the middle both spectra appeared strongly. The final fractions gave a brilliant spectrum of the second element, in which, however, the lines of the first element were still present. Even the oxide of the final mother-liquor gave with a strong spark the typical lines of Yb II. Even though the separation, in spite of every care, could not be regarded as complete, it seemed to me to be established beyond doubt that the preparation in the pure state of the two elements was at least within the bounds of possibility.

As from various experiments I had performed with the final mother-liquor, I had come to the conclusion that certain impurities, which could be detected in the liquor, had hindered the course of the separation. I next prepared the earth oxalate in the pure state. Nitric acid was added to the liquor till the tetracid ammonium oxalate was formed, when it was left to clear; the precipitate was afterwards drawn off and washed. The liquids were evaporated and ignited. The incombustible residue contained, besides much silicic acid, some alumina, a little iron, a trace of uranium, Cu, K, Na, &c. Also a small amount of ytterbium elements could be detected in it.

The earth oxalate precipitate was ignited. It gave about 8 grms. of oxide. This was dissolved in nitric acid, the clear liquid was evaporated, and the nitrate was cautiously fused till oxides of nitrogen began to appear. When cold the melt was turbid. When a little water was added, and it was warmed, it dissolved to a turbid liquid. On addition of some more water and boiling a white basic nitrate separated. After some time this precipitate was drawn off and dissolved in nitric acid. Sulphuretted hydrogen produced only a very small precipitate in the faintly acid solution. The filtrate was similarly precipitated with sodium thiosulphate. As this first precipitate showed the ytterbium elements in the spark spectrum it was again dissolved in hydrochloric acid, and cautiously re-precipitated with thiosulphate. This precipitate, which was small in proportion to the quantity used, consisted of almost pure thorium salt, as shown by spectral analysis.

In the principal quantity of the nitrate solution sulphuretted hydrogen produced only an almost inappreciable turbidity. From the mother-liquor of the thiosulphate precipitate the hydrates were next prepared, then they were dissolved in nitric acid, and added to the principal quantity. The earths contained in the nitrate solution I now subjected to other separation processes which seemed suitable for the separation of Yb I. But I was not successful. The small quantity of Yb II. which this preparation still contains is not in the least degree affected.

So then I returned to the original method of separation. As I had already observed that the separation is easier the less supersaturated the solution is, I now allowed the mother-liquor always to stand eight days over the crystals which had first separated. Repeated spectral examinations showed that the separation was satisfactorily effected. After repeated evaporation only a small quantity of mother-liquor was finally obtained; the oxides from it in the pure state gave in the visible part with an ordinary spark only the spectrum Yb I. The brightest lines of Yb II. could only be seen with a very strong spark, and even then there were only indications of them.

As I intended to repeat the separation processes later in order to obtain larger quantities of Yb I. I contented myself at first with only a few series. The final mother-liquor of the second and third series gave a product approximately resembling the first mother-liquor.

In order to prove whether there was another rare earth or another element between Yb I. and Yb II. I examined fractions 11, 12, and 13 thoroughly. These fractions contained about 100 grms. of oxide. With a layer of 60 mm. the colourless concentrated nitrate solution showed an indication of an absorption band in the red. However, this totally disappeared if the absorbing layer was rather less. The oxide of this fraction was white, and gave no flame spectrum. The spark spectrum was like that of pure ytterbium.

Sulphuretted hydrogen produced no precipitate, nor potassium sulphate, which proved that the sulphide forming elements were not present. The pure nitrate solution was finally again precipitated with acid ammonium oxalate, the

oxalate dissolved in ammonium oxalate with the addition of ammonia, and allowed to crystallise.

To prepare the element Yb II. in the pure state I made a new series from fractions 1 to 10 of the last series, and from the products obtained from the second working up process just described.

From some members of this series ammonium oxalate was removed, because they contained more of it than was necessary to dissolve the double salt.

The solution of the crystal fractions was always effected on the water-bath. The mother-liquor of the final fraction of each series was put aside, and the first fraction of the series always taken up with water. I allowed all fractions to crystallise for forty-eight hours. As soon as the first fraction contained but little ammonium oxalate, or none at all, thus either did not dissolve freely in water or was quite insoluble, it was put aside. Thus I obtained after about thirty series ten fractions very rich in earths. The first showed both Er and Tu in the absorption spectrum. The fourth fraction was pure white, like all the subsequent ones. They no longer showed an Er absorption spectrum. The fifth gave an oxide, the fairly concentrated nitrate solution of which (0·5 grm. oxide in 1 cc.) was perfectly colourless. The solution in a layer of 30 mm. showed only a faint absorption band in the extreme red. This band ascribed to thulium was lost in the later fractions.

The spark spectrum of these fractions, even with a very strong spark, gave none of the characteristic lines of Yb I. in the visible part of the spectrum. Only the lines peculiar to Yb II. appeared.

Thus the purity of the preparation had reached the limits of sensibility given by ocular observation. I now turned to the methods of investigation which would be most sensitive in the present case, *i.e.*, photographing the ultra-violet part of the spectra.

The developed plates showed the spark lines with extraordinary sharpness. By direct comparison of the spectrograms it could be shown (of course disregarding the lines common to both elements) that neither Yb I. nor Yb II. was quite pure.

I therefore determined to repeat the separation processes. With Yb II. this seemed to be fairly promising. For I had obtained a fairly large amount of this substance, large enough to render a new separation series possible. But I decided not to continue the work with Yb I., on account of the small quantity at my disposal. After I had left this very tedious work for some time I arranged the fractions rich in Yb II. in a new series.

Unfortunately my labours were not attended with success. In spite of numerous repetitions of the crystallisation process, in spite of many alterations of the method of separation, I did not succeed in separating Yb II. from Yb I. completely. In the spectrograms the well-marked lines of Yb I. always appeared. So I gave up further separation experiments. In considering the purity of the preparations it must be remembered that by taking photographs of the spectra the sensitiveness of the test may be increased to almost any desired extent. With the most sensitive plates it depends only upon the time of exposure whether a line is strongly marked on the plate or not. But if it is remembered that some lines of the two new elements act so strongly on the photographic plate that even with a fairly weak spark an exposure lasting only one second is sufficient to photograph the line clearly, it may easily be understood that with an exposure of 200 seconds or more the elements which are so difficult to separate do not appear to be free from one another.

I intend to repeat the separation process described in this article with more material than I had at my disposal, partly so that I may provide other investigators with sufficiently pure specimens for further measurements, and partly to find out whether by pushing the fractionation still further Yb I. can be obtained in a much purer state. It will take six or eight years to complete this tedious task.

As may be seen from the following description of the spark spectra of the two new elements, neither Yb I. nor Yb II. seems to be able to be further decomposed by the means now at our disposal.

As the decomposition of ytterbium into two new elements has been proved experimentally beyond doubt, it will be best to use the name ytterbium in the future only as a collective name, and to give the new substances new names. I suggest the name—

Aldebaranium (symbol Ad)

for the element called in the first part of this article Yb II., and resembling thulium or erbium, and—

Cassiopeïum (symbol Cp)

for the second element, called Yb I. in this article, the last in the series of the rare earths.

As regards the chemical properties of the two elements I can state them very shortly. The reactions are those of ytterbium. Chemically they cannot be distinguished from one another by any reactions, however carefully carried out. They form only one oxide which is stable at a red heat; it is a sesquioxide. All salts are derived from this. They are colourless if the acid is not coloured.

The Atomic Weights.

The atomic weights of the elements are very close.

Determinations performed according to Bunsen's method gave :—

For Aldebaranium.
(O = 16).

Analysis.	Oxide.	Sulphate.	Atomic weight.
I.	0·4181	0·6730	172·98
II.	0·5984	0·9634	172·88
III.	0·6173	0·9939	172·85
		Mean Ad	172·90

For Cassiopeïum.

I.	0·3716	0·5967	174·25
II.	0·3086	0·4956	174·19
III.	0·4026	0·6465	174·24
		Mean Cp	174·23

The following notes on these determinations may be added :—

The oxide of Analysis I. of aldebaranium was derived from an oxalate which had been obtained by repeated precipitation from strong nitric acid solution.

The oxides of Analyses II. and III. of Ad, as well as the oxides of the analyses of Cp, were obtained from a nitrate solution containing much ammonium nitrate and free from CO_2, which was repeatedly precipitated with ammonia free from CO_2.

The accuracy of the atomic weight determinations of these elements, apart from the magnitude of the atomic weight, were also affected unfavourably by the fact that the sulphates of these elements, on ignition, are very unstable as compared with other elements.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 2nd instant, Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, in the Chair. Viscount Ridley, Dr. William Hunter, and Mr. Andrew Bennie were elected Members. The Honorary Secretary reported the decease of the Rt. Hon. the Earl of Rosse, K.P., F.R.S., a Manager and Vice-President, of Professor Henri Becquerel, and Prof. E. Mascart, Hon. F.R.S., Honorary Members, and Resolutions of Condolence with the families were passed.

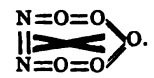
THERMO-CHEMICAL EQUIVALENCE AND THE THERMO-CHEMISTRY OF NITROGEN.

By J. C. THOMLINSON, B.Sc.

When I originally suggested, through the columns of the Chemical News, the numbers given for thermo-chemical equivalents of some of the elements, I was aware that other numbers might be obtained algebraically which would give results with equal approximation to those obtained by experiment.

I might quote one author on chemistry who says that "the prime object of the study (thermo-chemistry) is to obtain relative measurements of the chemical affinities inducing reactions," and state, further, that the behaviour of ozone and the oxides of nitrogen lead me to think that the numbers I gave might actually represent the thermal changes taking place.

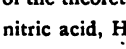
Having stated my belief in this possibility, I venture the following structural formula for nitrogen pentoxide:—



Theoretically, a compound with this structure would have an approximate heat of formation 12·504 cals., the experimental heat of formation being 13·000 cals., and this for solid nitrogen pentoxide.

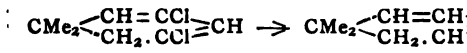
Another author I quote gives the heat of formation of nitrogen pentoxide (gaseous) zero, and solid as above.

As far as I can gather the gaseous pentoxide has not been isolated, and believing that the latent heats of liquids are, as a rule, less than that of water (80 cals.) from the liquid to solid state, I venture to think that this statement for gaseous nitrogen pentoxide need not detract from the correctness of the theoretical result I have given above.

Also for nitric acid,  , I find the calculated heat of formation 45·447 cals., as against 41·600 cals.—the experimental value.

THE STUDY OF HYDRO-AROMATIC SUBSTANCES.*

1 : 1-Dimethyl-Δ² : 4-dihydrobenzene and 1 : 1-Dimethyl-Δ² : 5-dihydrobenzene (Crossley and Renouf, *Journ. Chem. Soc.*, 1908, xciii., 629).—An account of the preparation of 1 : 1-dimethyl-Δ² : 4-dihydrobenzene (II.) by the reduction of 3 : 5-dichloro-1 : 1-dimethyl-Δ² : 4-dihydrobenzene (I.) with sodium in moist ethereal solution appeared in 1902 (Crossley and Le Sueur, *Journ. Chem. Soc.*, 1902, lxxxi., 821).



I. II.

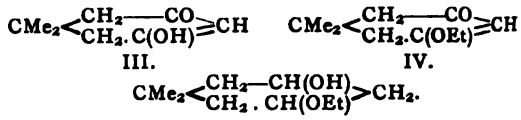
Shortly after the publication of this work, Harries and Antoni expressed doubt, not only as to the constitution, but also as to the composition of this hydrocarbon (*Annalen*, 1903, cccxxviii., 88). Their criticisms were, in part, answered at the time (*Ber.*, 1903, xxxvi., 2692), but it was considered desirable to try to prepare the substance by another method, in the hope that a comparison of the two specimens of the hydrocarbon would throw further light on the problem. This has now been done, with the result that the correctness of the earlier experiments and deductions are completely confirmed, allowing the following conclusions to be arrived at:—

1. The dimethyldihydrobenzene described by Crossley and Le Sueur is 1 : 1-dimethyl-Δ² : 4-dihydrobenzene, containing as impurity a very small amount of some oxygenated substance, probably a methoxy-compound.

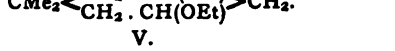
2. The hydrocarbon described in this report (dimethyldihydrobenzene, C. and R., see Note), and prepared by the elimination of two molecules of hydrogen bromide from dibromodimethylhexahydrobenzene, is a mixture, in approximately equal quantities, of pure 1 : 1-dimethyl-Δ² : 4-dihydrobenzene and pure 1 : 1-dimethyl-Δ² : 5-dihydrobenzene.

Note.—To avoid confusion as far as possible, the hydrocarbon described in this report, which is a mixture of pure 1 : 1-dimethyl-Δ² : 4-dihydrobenzene and of pure 1 : 1-dimethyl-Δ² : 5-dihydrobenzene, is referred to as dimethyldihydrobenzene (C. and R.), the 1 : 1-dimethyl-Δ² : 4-dihydrobenzene, prepared by Crossley and Le Sueur, as dimethyldihydrobenzene (C. and S.), and the supposed 1 : 1-dimethyl-Δ-² : 5-dihydrobenzene of Harries and Antoni (*loc. cit.*) as dimethyldihydrobenzene (H. and A.).

The starting-point in the synthesis was again dimethyldihydroresorcin (III.), which was first converted into its ethyl ether (IV.) (*Journ. Chem. Soc.*, 1899, lxxv., 775), and this by reduction with sodium in absolute ethyl-alcoholic solution into 3-hydroxy-5-ethoxy-1 : 1-dimethylhexahydrobenzene (V.):—

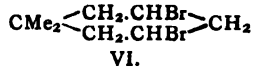


III. IV.

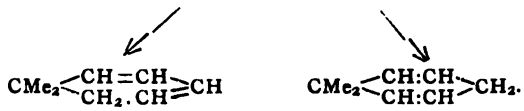


V.

The ethoxy-compound gives with fuming hydrobromic acid dibromodimethylhexahydrobenzene (VI.), which, when treaetd with quinoline, loses readily and completely two molecules of hydrogen bromide, with formation of dimethyldihydrobenzene, and this reaction takes place in two ways, giving rise to 1 : 1-dimethyl-Δ-² : 4-dihydrobenzene (VII.), or to 1 : 1-dimethyl-Δ-² : 5-dihydrobenzene (VIII.), in approximately equal amounts.



VI.



VII. VIII.

Table I. is a tabulated comparison of the properties of this hydrocarbon with those of the dimethyldihydrobenzene prepared by Crossley and Le Sueur. From this table the most important chemical properties of dimethyl-Δ² : 4-dihydrobenzene and dimethyl-Δ² : 5-dihydrobenzene would appear to be:—

1 : 1-Dimethyl-Δ² : 4-dihydrobenzene.	1 : 1-Dimethyl-Δ² : 5-dihydrobenzene.
Hydrobromide, $C_8H_{13}Br$, b. p. 83°/20 m.m.	Hydrobromide, $C_8H_{14}Br_2$, b. p. 137°/25 mm.
Nitrosochloride, m. p. 121°.	No nitrosochloride isolated.
Dibromide, $C_8H_{12}Br_2$, unstable liquid.	Tetrabromide, $C_8H_{12}Br_4$, m. p. 102°.
No nitro-derivatives obtainable.	Trinitro-*o*-xylenes, m. p. 71° and 115°.
Oxidation product, *as*-dimethylsuccinic acid.	Oxidation product, dimethylmalonic acid.

The criticisms of Harries and Antoni (*loc. cit.*) may then be replied to as follows:—

In the first place, these authors consider that the analyses given by Crossley and Le Sueur (*ibid.*) are midway be-

* Report of the Committee, consisting of Dr. E. Divers (Chairman), Prof. A. W. Crossley (Secretary), Prof. W. H. Perkin, Dr. M. O. Forster, and Dr. H. R. Le Sueur. Read before the British Association (Section B), Dublin Meeting, 1908.

TABLE I.

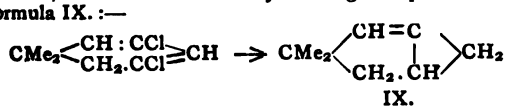
	Dimethyldihydrobenzenes.	
	C. and S.	C. and R.
Boiling-point	111°.	111·2°
Sp. gr. at 15°/15°	0·8153	0·8147
Index of refraction	1·4548	1·4535
Molecular refraction	60·217	60·251
Magnetic rotation	11·024	10·450
Colour with H_2SO_4	Blood-red, turning to violet-purple.	Blood-red, turning to violet-purple.
Fuming HBr	Monohydrobromide, b. p. 90·5°/36 mm.	Monohydrobromide, b. p. 83°/20 m.m.; dihydrobromide, b. p. 137°/25 mm.
Bromine	Dibromide, unstable liquid.	Dibromide, unstable liquid; tetrabromide, m. p. 102°.
NOCl	Nitrosochloride, m. p. 121°.	Nitrosochloride, m. p. 121°.
Nitrating mixture	No definite product.	Trinitro-*o*-xylenes, m. p. 71° and 115°.
Oxidation products	*as*-Dimethylsuccinic acid.	*as*-Dimethylsuccinic acid; dimethylmalonic acid.
Oxidation products of monohydrobromide	*as*-Dimethylsuccinic acid; ββ-dimethylglutaric acid; lactone of α-hydroxy-ββ-dimethyl glutaric acid.	*as*-Dimethylsuccinic acid; ββ-dimethylglutaric acid; lactone of α-hydroxy-3,3-dimethyl-glutaric acid.

tween those for a dimethyldihydro- and a dimethyltetrahydro-benzene. The mean of the two analyses quoted gave C = 88·30 and H = 11·27, and it is obvious from the annexed comparison that the numbers do not lie midway between the calculated figures, the differences in those found and calculated for C_8H_{14} being very large, and the errors in the wrong direction; for, as a rule, carbon is found too low and hydrogen too high, and not *vice versâ*. Moreover, if any appreciable quantity of dimethyltetrahydrobenzene had been present it could hardly have escaped detection, as on oxidation it would have yielded ββ-dimethyladipic acid, which presents no difficulty of isolation or identification.

Difference.	Calculated. C_8H_{14}.	Found.	Calculated. C_8H_{12}.	Difference.
+1·03	87·27	C = 88·30	88·88	−0·58
−1·46	12·73	H = 11·27	11·11	+0·16

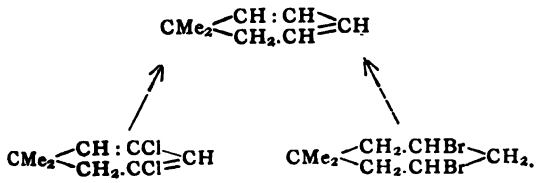
If the above-quoted figures for dimethyldihydrobenzene (C. and S.) are examined, it will be found that they add up to 99·57; and it is considered most probable that the discrepancy is due to oxygen present in the form of traces of a methoxy-compound.

Another suggestion made by Harries and Antoni is that dimethyldihydrobenzene (C. and S.) is not a true dihydrobenzene, but contains a trimethylene ring as represented in Formula IX.:—



IX.

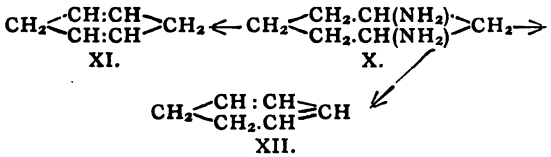
Altogether apart from the probable incapability of existence of such a body, it may be mentioned that the magnetic rotation of the hydrocarbon would have at once proved the presence of a trimethylene ring, and, moreover, there is now evidence that the same dimethyldihydrobenzene is produced by the reduction of dichlorodimethyldihydrobenzene, and by the removal of the elements of hydrogen bromide from dibromodimethylhexahydrobenzene.



This can only be explained if the substance be designated 1 : 1-dimethyl-Δ^2 : 4-dihydrobenzene, and hence the possibillty of a trimethylene ring being present in the hydrocarbon is precluded.

The whole question is further complicated by the fact that Harries and Antoni prepared a hydrocarbon, which they believed to be 1 : 1-dimethyl-Δ^2 : 5-dihydrobenzene also from dimethyldihydroresorcin as a starting-point; but, unfortunately, no information is given as to any derivatives which would enable a comparison to be made with the hydrocarbon having that undoubted structure described in this report; and it would appear that the present evidence is insufficient definitely to establish the constitution of Harries and Antoni's hydrocarbon. The single piece of evidence on which the supposed constitution of this latter substance is based is oxidation, whereby oxalic, a succinic, and malonic acids were obtained, none of which bodies afford any direct proof that the hydrocarbon contained the *gem*-dimethyl group.

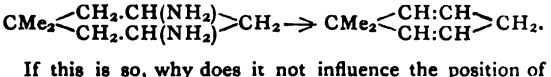
Nor is there any apparent reason for supposing that the elimination of ammonia from the cyclic diamine would take place in one way and one way only. Harries and Antoni have themselves shown that, when the phosphate of diaminohexahydrobenzene (X.) is submitted to dry distillation, ammonia is evolved in both of the possible directions, giving rise to a mixture of Δ^1 : 3-dihydrobenzene (XI.) and Δ^1 : 4-dihydrobenzene (XII.)—



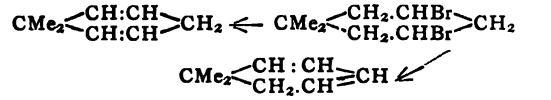
XI. X.

XII.

and they therefore conclude that "the *gem*-dimethyl group influences the position taken up by the double bonds"—



If this is so, why does it not influence the position of the double bonds when hydrogen bromide is eliminated from dibromodimethylhexahydrobenzene? For this is a



reaction strictly analogous to the reaction used by Harries and Antoni, and yet it takes place in both ways, giving rise to a mixture of the two possible hydrocarbons in equal

amounts. The presence of the *gem*-dimethyl group certainly does not exercise any apparent influence in this case, nor in the position of the double bond formed when 3-bromo-1 : 1-dimethylhexahydrobenzene loses the elements of hydrogen bromide (*Journ. Chem. Soc.*, 1906, xxxix., 1556).

Harries and Antoni state that 2 grms. of their dimethyldihydrobenzene gave, on treatment with a nitrating mixture, 0·6 grm. of trinitro-*m*-xylene, which might be regarded as a proof that dimethyldihydrobenzene (H. and A.) does not contain the *gem*-dimethyl group, for though many cases of the wandering of a methyl group in derivatives of *gem*-dimethyldihydroresorcin have been recorded (*Journ. Chem. Soc.*, 1904, lxxxv., 264; 1906, lxxxix., 875), no single instance has been observed in which a methyl group has wandered into anything but an ortho- position. Moreover, dimethyldihydrobenzene (C. and R.) undoubtedly contains dimethyl-$\Delta^{2:5}$-dihydrobenzene, and under the influence of a nitrating mixture it is this hydrocarbon which is converted into the two possible trinitro-*o*-xylenes.

(To be continued).

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

Part III.—Analysis of the Aluminium and Iron Groups, including Beryllium, Uranium, Vanadium, Titanium, Zirconium, and Thallium.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 221).

Confirmatory Experiments and References (continued).

*P. 51*a*, N. 1: *Test for Vanadate with* H_2S *in Alkaline Solution.*—To separate solutions containing 3 grms. NH_4Cl and 5 cc. NH_4OH (0·90) in a volume of 110 cc. were added 0·1, 0·3, and 0·5 mgrm. V as vanadate, and the mixtures were saturated with H_2S: in the experiments with 0·3 and 0·5 mgrm. V the solutions quickly became dark yellow, then reddish yellow, and finally pink; in that with 0·1 mgrm. V the solution finally became faintly pink, but the test was a poor one. Therefore, the limit of detectability under these conditions is 0·1 to 0·2 mgrm.

The experiment with 0·5 mgrm. V was repeated except that, instead of 5 cc. NH_4OH (0·90), 1, 2·5, and 10 cc. respectively were used: with 1 cc. and with 2·5 cc. NH_4OH the pink colour was scarcely noticeable; with 10 cc. a good colour was obtained as with 5 cc. NH_4OH, but only after passing in H_2S for a longer time. These experiments show that a fairly large excess of NH_4OH is necessary.

20 mgrms. V as Na_3VO_4 were added to 20 cc. NH_4OH (0·96) and H_2S passed in: the colour quickly became brown and slowly turned red, becoming after five or six minutes a deep cherry-red. The solution was divided into two parts. To the first was added an equal volume of water: the red colour disappeared in two or three minutes, but appeared again on re-saturating with H_2S. To the second part of the red solution was added an equal volume of NH_4OH (0·96): the colour faded slowly (in four or five minutes) but reappeared on passing in H_2S. These experiments show that the solution must be saturated with H_2S.

1 mgrm. V as Na_3VO_4 was dissolved (*a*) in 20 cc. NH_4OH (0·90), (*b*) in 20 cc. NH_4OH (0·96), (*c*) in 5 cc. NH_4OH (0·96) and 15 cc. water, (*d*) in 2 cc. NH_4OH (0·96) and 38 cc. water, (*e*) in 2 cc. NH_4OH (0·96) and 98 cc. water. Each solution was saturated with H_2S: a good colour was obtained in every case, showing that in the absence of ammonium salts the test may be obtained in any concentration of NH_4OH.

A solution containing 1 mgrm. V as Na_3VO_4 and 3 cc. HNO_3 (1·42) in 10 cc. was neutralised with NH_4OH (0·90) and 1 or 2 cc. in excess added: on saturating with H_2S a very faint colour was obtained. The experiment was repeated, except that the acid solution was first evaporated to dryness and ignited: a very good test was obtained, showing that the presence of ammonium salts interferes with test for vanadium.

0·5 mgrm. V as Na_3VO_4 was added to some HCl (1·20) and the mixture was evaporated twice nearly to dryness to reduce the vanadic acid to hypovanadic acid; 4 cc. HCl (1·12) were added and 96 cc. water; the mixture was neutralised with NH_4OH (0·90), an excess of 5 cc. was added, and H_2S passed in for ten to fifteen minutes: the solution darkened quickly, became reddish yellow, and finally pink; the colour was exactly the same as in the experiment described above with 0·5 mgrm. V as Na_3VO_4. The experiment was repeated with 25 mgrms. V, which was reduced to hypovanadic acid by long continued treatment of the hot solution with H_2S: the solution darkened and then became deep red very quickly; the colour was the same as in the experiment described above with 50 mgrms. V as Na_3VO_4.

1 mgrm. V as Na_3VO_4 was dissolved in 18 cc. water, 2 cc. 10 per cent NaOH solution were added, and the mixture saturated with H_2S: a deep red colour was finally obtained which was almost the same as that obtained when NH_4OH was used. The experiment was repeated with 20 cc. undiluted NaOH solution: the final colour was the same, but the solution remained colourless for a long time while the H_2S was being passed through it. Therefore the formation of the red compound does not depend on the presence of NH_4OH or $NH_4\times$ ion.

*P. 51*a*, N. 2: *Action of Acids on Sulphovanadate.*—0·5 mgrm. V as sodium vanadate was dissolved in 100 cc. containing about 3 grms. NH_4Cl; 5 cc. NH_4OH (0·90) were added, and the mixture saturated with H_2S: the pink solution was filtered, and then acidified in the cold with acetic acid, stirred, and filtered: a small black precipitate containing sulphur was obtained. This was dissolved by boiling with a little HNO_3 (1·20); the solutions were evaporated to about 2 cc. and 1 to 2 cc. 3 per cent H_2O_2 were added: an orange-yellow colour resulted, showing the presence of a small amount of vanadium. The experiment was repeated with sodium hypovanadate: the results were the same.

The experiment was repeated with 25 mgrms. V: on boiling the filtrate from the precipitate of vanadium sulphide it became blue and sulphur separated. To it 50 mgrms. Fe as $FeCl_3$ were added, and then an excess of NH_4OH: the mixture was filtered, the filtrate was evaporated to dryness, the residue ignited and dissolved in a very little HNO_3, and a few drops H_2O_2 solution were added: no colour appeared, showing that the vanadium in excess of 0·1 mgrm. had been carried down with the $Fe(OH)_3$. The sulphide and hydroxide precipitates were dissolved separately in HNO_3, H_2O_2 was added together with 20 to 30 cc. water, and the intensities of the colours compared: it was estimated that about 10 mgrms. V were precipitated as sulphide, and the remaining 15 mgrms. V with the $Fe(OH)_3$. The experiment was repeated with 50 mgrms. V, a larger amount of $FeCl_3$ being used: the results were similar, thus showing that under these conditions less than half the vanadium is precipitated as sulphide. In other experiments in which the volume was only 20 to 30 cc., and the relative proportion of ammonium salt to NH_4OH was smaller, 80 to 90 per cent of the vanadium was found to be precipitated as sulphide by acetic acid. Even when NaOH was used instead of NH_4OH, it was not possible to precipitate the sulphide completely.

The experiment with 50 mgrms. V described in the last paragraph was repeated, except that HCl was used to

* From the *Journal of the American Chemical Society*, xxx., No. 4.

acidify the cold solution: it was estimated that roughly one-half of the vanadium was precipitated as sulphide and the other half with the $Fe(OH)_3$. The last experiment was repeated, except that the mixture containing HCl was boiled for about one minute: somewhat less than half the vanadium was found in the HCl precipitate.

*P. 51*a*, N. 3: *Complete Precipitation of Vanadium by Ammonium Hydroxide in the presence of Iron.*—See preceding section, and C. E., G. D., Section 1.

*P. 51*a*, N. 4: *Pervanadic Acid.*—See Scheur (*Zeit. Anorg. Chem.*, 1898, xvi., 284); Pissarjewsky (*Zeit. Phys. Chem.*, 1903, xliii., 171 and 173; and 1902, xl., 368).

Action of H_2O_2 on Vanadic Acid.—In a series of six test-tubes, each of which contained 1 mgrm. V as Na_3VO_4, were placed 0, 0·5, 2, 5, 10, and 20 cc. H_2SO_4 (1·20); each solution was diluted to 20 cc. and 2 cc. 3 per cent H_2O_2 added: no change was observed in the tube with no acid: the solution became orange to orange-red in the remaining tubes, and the intensity of the colour increased greatly as the concentration of the acid increased. The experiments were repeated with HNO_3 (1·20): the results were the same. All the tubes were allowed to stand over night: no changes in the colours were observed. Ether was added to a number of the solutions and the mixtures shaken: the ether layer remained colourless.

To solutions containing 1 mgrm. V as Na_3VO_4 dissolved in 1 cc. HNO_3 (1·20) were added, (*a*) 1 cc. 3 per cent H_2O_2 and 8 cc. water, (*b*) 3 cc. H_2O_2 and 6 cc. water, and (*c*) 9 cc. H_2O_2: the first two solutions were orange-red, but the third solution was practically colourless, thus showing that a large excess of H_2O_2 spoils the test. To the third solution were added 3 cc. HNO_3 (1·20): the orange-red colour reappeared, thus proving again that the test is more easily obtained in the presence of a large excess of acid. To this solution was then added 7 cc. H_2O_2: the colour became much fainter.

The first series of experiments with varying amounts of HNO_3 (1·20) and a total volume of 20 cc. was repeated, except that 0·1 mgrm. V as Na_3VO_4 was used instead of 1 mgrm.: all the solutions remained colourless, showing that 0·1 mgrm. V cannot be detected in 20 cc. This series of experiments was repeated with 0·3 mgrm. V: a distinct yellow colour could be seen on looking down the tubes in the experiments with 5, 10, and 20 cc. HNO_3, a light colour with 2 cc. HNO_3, and none with 0·5 cc. This series was repeated with 0·5 mgrm. V: with 0·5 cc. HNO_3 the solution was colourless, with 2 cc. it was slightly yellow, and in the remaining experiments a slight but distinct orange-yellow colour was observed. Therefore the limit of detectability of vanadium with H_2O_2 in HNO_3 solution is about 0·5 mgrm. V in 20 cc., and the solution must contain at least 1 volume HNO_3 (1·20) to 3 volumes of water.

*P. 51*a*, N. 5: *Tungstic Acid dissolves in Acids when Phosphate or Arsenate is present.—Tech. Quart.*, 1903, xvi., 122.

Partial Precipitation of Tungsten on Acidifying a Solution of Sulphotungstate.—See *Tech. Quart.*, 1904, xvii., 253—5.

Action of H_2S on Tungstate dissolved in excess of NH_4OH.—50 mgrms. W as $(NH_4)_2WO_4$ and 3 grms. NH_4Cl were dissolved in 100 cc. water, 5 cc. NH_4OH (0·90) were added, and the mixture was saturated with H_2S: the solutions remained nearly colourless for over ten minutes, but finally became slightly yellow, probably owing to the formation of polysulphide.

Action of H_2O_2 on Tungstic and Molybdic Acids (see *Tech. Quart.*, 1904, xvii., 251). — 20 mgrms. Mo as $(NH_4)_2MoO_4$ were dissolved in 5 cc. HNO_3 (1·20) and 2 cc. H_2O_2 added: a lemon-yellow colour resulted. On account of the absence of an orange tint there is no difficulty in distinguishing between this colour and that of pervanadic acid.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

THE following are abstracts of papers received during the vacation, and published or passed for publication in the *Transactions*:—

164. "*The Use of the Micro-balance for the Determination of Electro-chemical Equivalents and for the Measurement of Densities of Solids.*" By OTTO BRILL and CLARE DE BRERETON EVANS. (*Trans.*, 1908, 1442; this paper was taken as read at the meeting of May 21st, 1908).

By the use of the micro-balance the electro-chemical equivalent of copper is found to be 31·95, and that of antimony 40·9, the amount of metal used being from one-third to one-half of a mgrm. The instrument may also be employed for determining the densities of solids which can be obtained in regular form, and thus measurable with a microscope. Results agreeing with those recorded by previous investigators were obtained by using about half a mgrm. of the substances selected.

165. "*The Preparation of Disulphides.* Part III. *The Nitrobenzyl Disulphides.*" By THOMAS SLATER PRICE and DOUGLAS FRANK TWISS. (*Trans.*, 1908, 1401).

The authors have prepared o-, m-, and p-*nitrobenzyl disulphides* by the action of alkali on solutions of the corresponding nitrobenzyl thiosulphates (compare Price and Twiss, *Trans.*, 1908, xciii., 1395). The solution of the complex thiosulphate is obtained in each case by boiling together an aqueous solution of sodium thiosulphate and an alcoholic solution of the nitrobenzyl chloride. The most satisfactory alkali for the subsequent action is sodium carbonate. The *o*-, *m*-, and *p*-disulphides obtained melt at 109·5°, 103°, and 126·5° respectively.

166. "*The Relation between Unsaturation and Optical Activity.* Part III. *Optically Active Salts of Acids containing Adjacent Unsaturated Groups.*" By THOMAS PERCY HILDITCH. (*Trans.*, 1908, 1388).

The *brucine* and *cinchonine* salts of anthranilic and salicylic acids, and of the acetyl and benzoyl derivatives of these acids, have been prepared and measured polarimetrically, and it is found that, in general, as the number of contiguous unsaturated groups increases, the effect on the optical rotatory power of the alkaloids also increases. Further evidence has been obtained as to the effect on the optical activity of a compound when two unsaturated groups are situated at varying degrees of proximity to each other in the molecule.

167. "*The Constitution of Glucose Derivatives.* Part I. *Glucose-anilide, -oxime, and -hydrazone.*" By JAMES COLQUHOUN IRVINE and ROBERT GILMOUR. (*Trans.*, 1908, 1429).

The behaviour of some glucose derivatives on methylation has been studied with a view to distinguishing between compounds produced from the aldehydic form of the sugar and those derived from the γ-oxidic isomerides. Glucoseanilide, when fully alkylated by the silver oxide reaction, is converted into tetramethyl glucoseanilide (m. p. 132—134°), identical in every respect with the compound previously obtained by condensing tetramethyl glucose with aniline. This result shows that glucoseanilide only contains four hydroxyl groups capable of methylation, and that the compound possesses the γ-oxidic linking characteristic of reducing sugars and glucosides. The compound should thus exist in two stereoisomeric forms (α- and β-), and evidence has been obtained that this is the case. When prepared by condensing glucose and aniline at +6°, a dextrorotatory variety is formed, which, in solution, is converted into the lævorotatory α-isomeride (α_D 20 − 52·3°), thus giving rise to extensive mutarotation. The same phenomenon has also been observed in the case of tetramethyl glucoseanilide in methyl-alcoholic solution.

Glucoseoxime behaves similarly towards the alkylating

mixture of silver oxide and methyl iodide, as pentamethyl glucoseoxime was obtained as the product of maximum methoxyl content. This compound is tetramethyl glucoseoxime methyl ether, as by hydrolysis tetramethyl glucose is produced. From this result it is deduced that glucoseoxime, in addition to reacting as a normal oxime, is equally capable of behaving as a γ-oxide.

Similar methods when applied to glucosephenylhydrazone led to no definite result, owing to extensive oxidation taking place.

168. "*The* cis-*and* trans-*Modifications of* 1-*Methyl*cyclo*hexan*-2-*ol*-4-*carboxylic Acid and their Conversion into* 1-*Methyl*-Δ^1-cyclo*hexene*-4-*carboxylic Acid.*" By Andrew Norman Meldrum and William Henry Perkin, Jun. (*Trans.*, 1908, 1416).

The authors have reduced 2-hydroxy-*p*-toluic acid by sodium and alcohol, and find that the product of reduction is readily converted into 1-*methyl*-Δ^1-cyclo*hexene*-4-*carboxylic acid*, $CMe\leqslant\begin{matrix}CH—CH_2\\CH_2\cdot CH_2\end{matrix}>CH.CO_2H$, by treatfirst with hydrobromic acid and then with diethylaniline.

169. "*Studies on the Viscosity and Conductivities of some Aqueous Solutions.* Part I. *Solutions of Sucrose, Hydrogen Chloride, and Lithium Chloride.*" By W. Heber Green.

The viscosity of sucrose solutions at 18° and 25°, the viscosity and conductivity of hydrochlotic acid at 25°, and of lithium chloride solutions at 18° and 25°, have been measured over ranges of concentration limited only by the solubilities of each substance.

The densities of lithium chloride solutions have been re-determined and compared with the results obtained by previous observers.

The viscosity results obtained show that various formulæ which have been suggested do not at all accurately represent the connection between viscosity and concentration, especially when concentrated solutions are examined. The most concordant result was obtained by using an expression of the form $\eta/\eta_0 = Av/w$, where v = volume of solute, and w = volume of solvent, present per unit-volume of solution.

170. "*Studies on the Viscosity and Conductivity of some Aqueous Solutions.* Part II. *Mixtures of Solutions of Sucrose and Lithium Chloride: a Contribution towards the Elucidation of the connection between Ionic Mobility and the Fluidity of the Solution.*" By W. Heber Green.

The conductivity and viscosity of aqueous solutions have been measured at 25° of three series of mixtures of lithium chloride and sucrose, in which the ratio of the number of lithium chloride molecules to the number of sucrose molecules was 1 : 0·2, 1 : 2·0, and 1 : 20 respectively.

By extrapolation from the results for these mixtures the conductivity and viscosity values have been calculated for a series in which the above ratio was 1 : ∞. In this way the variations in the ionic mobilities at infinite dilution of the salt, concomitant with change of fluidity due to the presence of sugar in solution, were rendered free from some, is not all, disturbing factors.

The curves obtained show that the ionic mobility is not simply proportional to the physical fluidity of the solution, but more nearly to the two-thirds power in the case of lithium chloride and sucrose; with other ions, and other molecules giving rise to viscosity, the exact function varies, and presumably depends on the relative molecular sizes.

With reference to Hartley, Thomas, and Applebey's use of pyridine as an agent for producing viscosity, the author maintains that Stokes's theorem does not hold for a body of molecular size migrating through a medium composed of bodies comparable in size to itself, and, further, that there is no direct evidence for the presence of an aqueous envelope permanently attached to each ion.

No trustworthy method has as yet been established whereby the true ionisation coefficients of a salt can be determined except in solutions of extreme dilution.

171. "*The Reduction of Refractory Oxides by Carbon.*" By Harold Cecil Greenwood. (*Trans.*, 1908, 1483).

A method has been devised for measuring with an accuracy of about ± 10° the temperatures of reduction of oxides by carbon, the apparatus being suitable for use up to at least 2000°. The reduction temperatures of the oxides of chromium, manganese, uranium, silicon, tungsten, zirconium, and thorium have been measured, and are all found to be far below those previously estimated.

Commencement of the reaction occurs sharply, but the progress of the reduction depends greatly on the intimacy of admixture. The nature of the carbon employed, as also the physical condition of the oxide, exerts a marked influence on the temperature of reduction.

The reactions, so far as the oxides mentioned above are concerned, are found to occur directly between the carbon and the oxide, and are not due to thermal dissociation.

172. "*The Production of Ferro-alloys.*" By Harold Cecil Greenwood. (*Trans.*, 1908, 1496).

This investigation is an extension of the work described in the preceding paper. In the case of some of the oxides studied, for example, silica and manganous oxide, it is found that a "catalytic" facilitation of the reduction takes place when metals such as iron, manganese, and copper are present. This confirms observations made in the technical preparation of ferro-alloys under conditions which are unsuitable for the isolation of the pure metal. The lowering of the reduction temperature appears to be due to mutual chemical affinity of the two metals, and also in some cases to the alloy being more fusible than the refractory metal; no evidence is forthcoming to show that reduction is assisted by the heat of reaction of ferric oxide and carbon when this oxide is added instead of metallic iron.

173. "*The Rapid Electro-analytical Deposition and Separation of Metals.* Part II. *Antimony and Tin. The Employment of a Diaphragm.*" By Henry Julius Salomon Sand. (*Trans.*, 1908, 1572).

By means of the apparatus already described, antimony has been estimated in sulphuric acid solutions and suspensions in the presence of hydrazine sulphate, and also in solutions containing chlorides. A high temperature is maintained, and the potential of the cathode is kept under control.

Tin has been deposited from stannic solutions containing a small amount of free sulphuric acid and a sufficiency of oxalic acid at a temperature near the boiling-point.

Antimony has been separated from tin. Any *b*-stannic acid which may be present must first be converted into the *a*-form. The electrolyte consists of equal volumes of sulphuric acid and water; a small amount of hydrazine sulphate is added, and the temperature maintained at a little above 100°. The maximum auxiliary voltage is 0·65.

The tin is determined electrolytically in the remaining liquid after neutralisation with ammonia and the addition of oxalic acid. After the greater part of the metal has been deposited, it is necessary to add hydrochloric acid and hydroxylamine hydrochloride in order that the electrolyte may be exhausted; a high temperature is maintained. A considerable number of analyses have been carried out in which the combined metals, taken in varying ratios, weighed approximately 1 grm. The time for the deposition of the antimony was usually about twenty minutes, and that for the tin about eighty minutes.

An apparatus which allows the employment of a diaphragm in rapid electro-analysis has been constructed, and tested in the determination of copper and its separation from iron.

174. "*The Measurement of a Homogeneous Chemical change in a Gas.* (*The Thermal Decomposition of Ozone.*)" By Herbert Edmund Clarke and David Leonard Chapman. (*Trans.*, 1908, 1638).

It has been commonly supposed that slow pyrogenic changes in gases have their seat principally, if not entirely,

on the walls of the enclosing vessel. The authors have succeeded in demonstrating that this conclusion is not true in the case of the decomposition of ozone. In vessels of German glass, of which the ratio of surface to volume is not too large, the rate of decomposition of ozone at 100° is independent of the area of the surface of the globe, a fact which proves that the change is a homogeneous one.

175. "*The Proteins of Egg-yolk.*" By Robert Henry Aders Plimmer. (*Trans.*, 1908, 1500).

In addition to the protein, vitellin, which contains 1 per cent of phosphorus, and which is insoluble in water, egg-yolk contains a second protein, which contains 0·1 per cent of phosphorus, and which is soluble in water and coagulable by heat.

It can be prepared either by extracting the yolks, after treatment with ether, with a large volume of water, and heating to boiling in the presence of a small quantity of acetic acid, or by boiling the water used for precipitating the vitellin; this is obtained by dissolving the yolk in 10 per cent sodium chloride solution, extracting with ether, and precipitating with 20 volumes of water. The second method gives a purer preparation of this protein.

This protein differs from ovalbumin in its content of nitrogen as also in the amounts of the various nitrogen constituents contained in it when determined by Hausmann's method, so that it is not identical with this protein; its behaviour towards ether also indicates this. The various amounts in which the nitrogen constituents are present in the protein are very similar to those in vitellin, and suggests a close relationship between these proteins in egg-yolk; hence the name of *livetin* is suggested for this new protein.

176. "*The Conductivities of the α-Oximino-fatty Acids.*" By John Kenneth Harold Inglis and Lottie Emily Knight. (*Trans.*, 1908, 1595).

The authors have prepared specimens of the first four of these acids by the action of nitrosulphonic acid on aceto-acetic ester and its derivatives. The conductivities agree fairly closely with those measured by Hantzsch and Miolati. Measurements of the conductivities of the sodium salts and comparison of the acids with one another have indicated that the acids are not all of the same configuration, but that oximinopropionic acid is different from the others. The authors have also found evidence of the existence of two forms of α-oximino-butyric and -valeric acids.

177. "*Studies of the Perhalogen Salts.*" (Part II.). By Charles Kenneth Tinkler. (*Trans.*, 1908, 1611).

The formation of periodides has been further investigated by means of the ultra-violet absorption spectra of solutions of iodine containing a halogen salt. The iodine additive compounds of trimethylsulphine iodide, bromide, and chloride, which may be either (I.) $(CH_3)_3\overset{(IV.)}{S}X\cdot I_2$ or (II.) $(CH_3)_3\overset{(VI.)}{S}\begin{matrix}X\\ I\\ I\end{matrix}$ (X = I, Br, or Cl), gave spectra which are identical with those of solutions of iodine containing potassium iodide, bromide, and chloride respectively. This agreement shows that these substances contain the true perhalogen groups, and affords evidence in favour of Formula I.

The formation of perbromides (by the addition of a bromide to bromine water) has also been investigated by the spectroscopic method, and evidence obtained of the existence of these substances in a large number of cases. Evidence was also obtained of the formation of bromine additive compounds of chlorides.

178. "*The Formation of Ethers from Compounds of the Benzoin Type.*" By James Colquhoun Irvine and David McNicoll. (*Trans.*, 1908, 1601).

The authors find that the methyl ethers of benzoin should always be prepared by the silver oxide reaction, as by the use of Fischer's process of methylation the yields obtained are poorer, and in some instances furan derivatives are formed in quantity. In the case of anisoin, no methyl ether is produced in the latter reaction, which gives rise to the *tetramethoxyl* derivatives of α-methoxy-αδ-dibenzoyldibenzyl (m. p. 218—219°) and of 2-hydroxy-3 : 5-dimethoxytetraphenyltetrahydrofuran (m. p. 263—265°).

The hydrochloric acid method can also be applied to *o*-dimethoxybenzoin only within a limited range of concentration of the acid, as the benzoin readily undergoes molecular rupture to give *o*-methoxybenzoic acid. When the same process is applied to furoin, no methylation takes place, condensation products containing five furyl residues and analogous to those obtained from ordinary benzoin being produced. On the other hand, ethyl ethers of benzoins are comparatively readily prepared by Fischer's method, and no furan derivatives are formed.

In the course of the work, several benzoin ethers have been prepared by the silver oxide method, and the compound described by Limpricht and Jena as "ethyl benzoin" has been examined, and shown to be a mixture of hydrobenzoin and *iso*hydrobenzoin.

179. "*Aromatic α-Disulphones.*" By Thomas Percy Hilditch. (*Trans.*, 1908, 1520).

Aromatic α-disulphones, previously obtained only by the interaction of an aromatic sulphonyl chloride and the sodium salt of an aromatic sulphinic acid, have been prepared in yields varying from 17 to 40 per cent by the oxidation of sulphinic acids by potassium permanganate in glacial acetic acid solution. *Diphenyl, di-p-phenetyl, di-p-tolyl*, and *di-p-xylyl α-disulphones* have been thus obtained as well-crystallised stable substances of high melting-point; they are decomposed slowly by strong ammonia into a mixture of sulphonamide and sulphinate, and by warm aqueous potassium hydroxide into the corresponding sulphonates and sulphinates.

180. "*The Action of Bromine on β-Hydrindone.*" By Norman Allen Creeth and Jocelyn Field Thorpe. (*Trans.*, 1908, 1507).

The bromination of β-hydrindone may be effected by the action of bromine in chloroform solution, all four hydrogen atoms of the hydrindene ring being replaced by the halogen. A *monobromo-* and a *dibromo*-derivative may also be prepared by using the calculated quantity of bromine, but *ααγ*-tribromo-β-hydrindone cannot be produced in this way, since, when the amount of bromine required for its formation is employed, an equimolecular mixture of *αγ*-dibromo-β-hydrindone and *αααγγ-tetrabromo-β-hydrindone* is alone formed. The monobromo- and dibromo-derivatives are neutral substances insoluble in alkalis, showing, therefore, that the combination of the halogen with the α-carbon atom of β-hydrindone does not transform the compound into a derivative of hydroxyindene. The action of alkalis on the various bromo-derivatives has been investigated, but only in the case of *ααγγ*-tetrabromo-β-hydrindone has a well-defined product been obtained. In this instance, through a curious fission of the hydrindene ring, the product consists of phthalidecarboxylic acid.

181. "*On Polymorphism, with especial reference to Sodium Nitrate and Calcium Carbonate.*" By William Barlow and William Jackson Pope. (*Trans.*, 1908, 1528).

In continuation of their previous work, the authors discuss the relation between the crystalline form and the chemical constitution exhibited in connection with a number of well known cases of polymorphism.

182. "*The Oxidation of Phosphorous Acid by Iodine.*" By Bertram Dillon Steele.

In continuation of the investigation of the reaction between iodine and hypophosphorous acid, the velocity of interaction of iodine and phosphorous acid has been studied. It has been found that two reactions of essentially

different character occur between these reagents, one of which preponderates in more strongly acid solution, and the other, in the presence of feeble acids, such as acetic and boric acids.

The former of these reactions is catalytically accelerated by the presence of hydrogen ions, and the velocity is proportional to the concentration of phosphorous acid (or of the phosphorous ion) and to the square root of the concentration of the iodine.

The conclusion is drawn that reaction takes place between phosphorous acid molecules (or ions) and iodine atoms, the latter being supplied in extremely small but sufficient quantity by the dissociation of the iodine molecules. A slight apparent retardation of the reaction velocity by the iodine is probably due to the incompleteness of the hypothesis which has been suggested to explain the experimental results.

The second reaction, which occurs in the presence of salts, such as sodium acetate, bicarbonate, or biborate, is so complex that the attempt to formulate a hypothesis explanatory of the reaction has been abandoned. The reaction is accelerated by the presence of phosphorous acid, and of sodium acetate, and retarded by the presence of iodine and of potassium iodide.

183. "*The Direct Interaction of Magnesium and Alkyl Halides.*" By JAMES FREDERICK SPENCER and MARY S. CREWDSON.

It has been found that all alkyl halides react directly with magnesium. The halides of hydrocarbons lower in the series than butane only react when heated in a sealed tube, but, with the exception of the chlorides, which, so far as has been investigated, appear always to require heating in a sealed tube, all the halides of hydrocarbons higher in the series than butane react with magnesium when the two substances are heated together in a flask. The products are magnesium alkyl halides and hydrocarbons containing twice as many carbon atoms as the original halide. In the reactions taking place in sealed tubes, unsaturated hydrocarbons, of both the acetylene and the olefine series, together with much hydrogen, are also found, in addition to the above-mentioned compounds. Methyl bromoacetate also reacts directly with magnesium, giving good yields of methyl acetate and dimethyl succinate, and aromatic chloro-derivatives react with magnesium when heated with it in a sealed tube, yielding the corresponding hydrocarbons in good yield.

(To be continued).

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NOTICES OF BOOKS.

Laboratory Arts. By GEORGE H. WOOLLATT, Ph.D., F.I.C. London, New York, Bombay, and Calcutta: Longmans, Green, and Co. 1908.

THE author of this book has had special opportunities, during the summer courses for science teachers held in Ireland and Yorkshire, of finding out at first hand what problems in the construction and repair of all kinds of apparatus are most generally met with in laboratories, and he has turned his opportunities to the best account in the preparation of the book. It describes the use of tools for wood, metal, and glass work, and gives directions for the construction of various pieces of apparatus, some of them fairly complicated. Indeed, in some cases, more especially in glass work, it would seem that the cost of the finished article was out of proportion to the time and trouble which would have to be expended in its preparation in the laboratory. The sections on wood and metal work contain many suggestions which might be adopted in manual training classes, for which simple apparatus provides interesting and not unduly difficult exercises. In large laboratories at any rate, the value of the general information relating to the care of bench tops, the labelling of bottles, electrotyping, &c., which is given in the fourth section, would very soon repay the cost of the book.

Post's Chemisch-Technische Analyse. ("Post's Chemical Technical Analysis"). Edited by Prof. BERNHARD NEUMANN. Vol. II., Part III. Braunschweig: Friedrich Vieweg und Sohn. 1908.

THE third part of the second volume of this text-book contains comprehensive accounts of practical methods of analysing fertilisers, ethereal oils, tobacco, rubber, explosives, tanning materials, and some other substances. Each chapter has been prepared by an expert who has a thorough knowledge of the subject entrusted to him, and only the most modern and trustworthy methods find a place in the book. Both as a work of reference and as a laboratory guide "Post's Chemisch-Technishe Analyse" has every good feature to recommend it, and no scientific library should be regarded as complete unless it includes a copy of the third edition.

CORRESPONDENCE.

THE WAVE THEORY OF MOTION.

To the Editor of the Chemical News.

SIR,—I have read Mr. John McDowall's paper on the above subject in the CHEMICAL NEWS of October 30 (vol. xcviii., p. 213) with very great interest. By a line of reasoning different to that employed by him, I had already concluded that, granting the objective reality of time and space, it was not improbable that time is the "fourth dimension," and that the whole of the physical universe is moving in this timal direction.

On Friday, April 3, 1908, I had the honour of reading to the Polytechnic Mathematical Society a paper in which this view was expressed. In this paper I enunciated what I believe to be a new mathematical proof of the real existence of not only a fourth but an infinity of dimensions, providing that space itself is real. This proof will be found very briefly stated in a letter to the editors of *Knowledge and Scientific News* (vol. v., p 157, July, 1908). In the paper above mentioned I also applied this proof to time, and pointed out that time possessed the properties expected of a fourth dimension. This view, namely, that time is the "fourth dimension," is, however, not an original one, but it has generally been accompanied by the assumption that matter

is itself four-dimensional and extends through time, and that it is our consciousness that travels in a timal direction. In the above mentioned paper I rejected this hypothesis and suggested as more likely that the whole physical universe is rushing through time, as this would, at least so it appears, explain why time appears to us to be so different from length in space, and also why we have no consciousness of higher dimensions. I did not, however, mention these speculations in the letter to the editors of *Knowledge and Scientific News*, but about a month or two ago I prepared a revised essay on the subject which has since been pending publication.

In conclusion, I am most happy that Mr. McDowall's line of reasoning regarding the nature of motion should have confirmed my speculations regarding the fourth dimension, and I would be glad, if he should care to read it, to forward Mr. McDowall a copy of my original paper, in which this particular view is, I believe, first expressed, as the publication of the revised paper may be delayed for some time.—I am, &c.,

H. STANLEY REDGROVE.

The Polytechnic, Regent Street, W.,
October 30, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xli., No. 13, 1908.

Decomposition of Chromic Acid by Hydrogen Peroxide.—E. H. Riesenfeld.—The reaction between hydrogen peroxide, potassium bichromate, and sulphuric acid occurs according to the equation—

$$K_2Cr_2O_7 + 7H_2O_2 + 4H_2SO_4 = = Cr_2(SO_4)_3 + K_2SO_4 + 11H_2O + 5O_2,$$

when excess of hydrogen peroxide is present. The concentration of the sulphuric acid does not affect the reaction until the strength is 2·5 N, when some Caro's acid is formed, and gives up oxygen much quicker than hydrogen peroxide. When excess of chromic acid is present the equation is—

$$K_2Cr_2O_7 + 3H_2O_2 + 4H_2SO_4 = = Cr_2(SO_4)_3 + K_2SO_4 + 7H_2O + 3O_2,$$

and again the reaction is independent of the concentration of the sulphuric acid until the strength of the latter has been increased above 2·5 N. These two reactions of hydrogen peroxide with Cr_2O_7 may be represented by the ionic equations. I. In presence of excess of hydrogen peroxide,

$$Cr_2O_7'' + 7H_2O_2 = 2CrO_8''' + 4H\cdot + 5H_2O,$$
$$2CrO_8''' + 12H\cdot = 2Cr\cdots + 6H_2O + 5O_2.$$

II. In presence of excess of chromic acid,—

$$Cr_2O_7'' + 3H_2O_2 = 2CrO_5' + 3H_2O,$$
$$2CrO_5' + 8H\cdot = 2Cr\cdots + 4H_2O + 3O_2.$$

The author's researches show that chromic acid and chromates are never reduced by hydrogen peroxide directly to chromic oxide salt, but that perchromates are always formed, and these in acid solution give up oxygen and form chromic salts.

Preparation and Constitution of Sulphoxides and Disulphoxides.—O. Hinsberg.—Sulphides are converted into sulphoxides and disulphides into disulphoxides by the action of hydrogen peroxide in the cold. The formula of disulphoxide might be R.SO.SO.R or R.S.SO_2R. Since, however, the divalent sulphur of the sulphide is oxidised by hydrogen peroxide at any rate at room temperature to the SO group only, for sulphoxides and not sulphones are formed, the disulphides are in all probability converted into oxidation products of formula R.SO.SO.R, tetravalent sulphur being formed. The chemistry of sulphur gives us no reason for thinking that the group .SO.SO. is labile, but rather that it is stable, and hence the disulphoxides have in all probability the formula R.SO.SO.R. When disulphoxides are boiled in alcoholic solution with alkali large quantities of disulphides are formed. This reaction is very easily explained if it is assumed that alkalis transform the disulphoxides into unstable thioesters, which then yield disulphides, R.SO.SO.R $\rightarrow$ R.S.SO_2.R $\rightarrow$ R.S.S.R.

Sodium Compound of Fluorene.—R. Weissgerber.—When sodamide is introduced into fluorene heated to 120°, ammonia is evolved, and sodium fluorene separates out as a brownish yellow mass. When the mixture is heated to about 150° and allowed to cool, a brownish yellow crystalline mass is formed. The compound has the formula $\begin{matrix}C_6H_4\\C_6H_4\end{matrix}\!>\!CH.Na$. The preparation of this substance can conveniently be used to separate fluorene from coal-tar mixtures on a large scale. None of the hydrocarbons which accompany fluorene in coal-tar, acenaphthene, phenanthrene, &c., form a sodium compound. The fluorene-sodium can be separated by mechanical means from the mixture, and the fluorene recovered by decomposing it with water, distilling off the raw fluorene, and allowing it to crystallise.

Ultra-violet Fluorescence in Cyclic Compounds.—H. Ley and K. von Engelhardt.—The authors have investigated the mono- and di-substitution products of benzene with reference to their ultra-violet fluorescence, using Stark's method. Alcoholic solutions of concentration 0·005 normal were employed. The most important results obtained were as follows:—*Hydrocarbons.*—Toluene and propyl benzene have a much stronger fluorescence than benzene. The four bands characteristic of benzene disappear. *Amino Compounds.*—Aniline shows a much stronger fluorescence than benzene, also β-naphthylamine fluoresces more strongly than aniline. Dimethylaniline shows distinct fluorescence, which, however, is less than that of aniline. *Hydroxyl Compounds.*—The hydroxyl and methoxyl groups increase fluorescence; thus phenol and anisol exhibit the phenomenon very strongly. Benzoic acid fluoresces feebly, oxybenzoic acid more strongly; of the isomeric oxybenzoic acids the ortho-compound shows the strongest fluorescence and the para-compound the weakest. *Halogen Compounds.*—The halogens act like the carboxyl group, and their action is more marked the higher the molecular weight is. *Benzonitrile and Benzamide.*—Benzonitrile fluoresces strongly, while benzamide does not exhibit the phenomenon. *Nitro Compounds.*—The nitro group usually hinders fluorescence.

Picrates.—O. Anselmino.—When picric acid or yellow ammonium picrate is heated in 10 per cent ammonia solution to 80°, blood-red crystals are formed containing one molecule of acid to one molecule of ammonia. When these red crystals are re-crystallised they yield only the usual light yellow needles of ammonium picrate. The author has not yet decided whether the red colour of the first crystals is due to an impurity which has arisen from the action of hot alkali on picric acid.

Iodopentamminocobaltic Salts.—A. Werner.—The compounds obtained by J. Sand and G. Böckmann by the action of iodine on black nitrosopentamminocobaltic salts belong to the hitherto unknown iodopentamminobaltic series, and have the general formula $\left[Co^{I}_{(NH_3)_5}\right]X_2$. Aquopentamminocobaltic iodide, $\left[Co^{OH_2}_{(NH_3)_5}\right]I_3$, when heated to 80°, readily loses water, yielding a brown difficultly soluble product which, with ammonium nitrate, gives a good yield of green iodopentammincobaltic nitrate. This shows, beyond doubt, that the salts belong to the

iodopentammin series. The colours of the halogen pentammincobaltic salts are as follows:—

$\left[Co^{Cl}_{(NH_3)_5}\right]X_2$, Purple. $\left[Co^{Br}_{(NH_3)_5}\right]X_2$, Violet-blue. $\left[Co^{I}_{(NH_3)_5}\right]X_2$. Green.

Relations between Nickel and Hydrogen.—M. Mayer and V. Altmayer.—The authors have investigated the absorption of hydrogen by nickel at constant volume, the pressure of the hydrogen and the temperature being altered. The pressure was increased from one-fifteenth to four-fifths of an atmosphere and the temperature from 360° to 560°, and the volume of hydrogen absorbed by the nickel was observed. It was thus found that the graphs obtained were straight lines corresponding to Henry's law; *i.e.*, the masses of gas taken up are directly proportional to the pressure. Thus the system nickel-hydrogen is bivariant.

Methane Syntheses with Calcium Hydride.—M. Mayer and V. Altmayer.—Calcium hydride may be prepared by passing hydrogen over metallic calcium in turnings at a temperature which is gradually raised from 270°. The hydride thus obtained is grey in colour and can easily be powdered. It falls to a powder in air, and is decomposed by water and dilute acids, giving hydrogen. When carbon monoxide is led over the hydride at temperatures from 400° to 800°, methane is formed, and a black residue mixed with a little grey substance is left behind. This residue smells slightly of acetylene. When it is treated with water, gas is energetically evolved, and a strong smell, first of acetylene and then of ammonia, is noticed. The reaction seems to be as follows:—

$$3CaH_2+3CO=CH_4+3CaO+2C+H_2,$$
$$CaH_2+2C=CaC_2+H_2.$$

When carbon dioxide replaces the monoxide in this experiment the reaction is represented by the equation $2CO_2+4CaH_2=CH_4+4CaO+C+2H_2$. The residue resembles the former residue in appearance, but has no smell. When water is added to it no gas is evolved till the mixture is heated, then a slight smell of acetylene is noticed. With dilute hydrochloric acid carbon dioxide is evolved. When carbon acts on calcium hydride some methane is evolved above 270°. The residue is black, and smells of acetylene. It gives large quantities of gas when treated with cold water, and strong smells, first of acetylene and then of ammonia, are observed. The interior of the flask in which the residue is treated with water becomes very hot.

Action of Hydrazine Hydrate on Bisdiazoacetic Ester.—Th. Curtius and Eugen Rimele.—The first product of the action of hydrazine hydrate on bisdiazoacetic ethyl ester is bisdiazoacetic acid ethyl ester hydrazide, $COOC_2H_5.C\overset{N\text{---}N}{<_{NH\text{---}NH}>}C.CO.NH.NH_2$. The further action of the hydrazine hydrate converts this compound into bisdiazoacetic acid bishydrazide, $NH_2.NH.CO.C\overset{N\text{---}N}{<_{NH\text{---}NH}>}C.CO.NH.NH_2$. Thus bisdiazoacetic ester reacts with hydrazine hydrate like terephthallic acid ester, which also yields an ester hydrazide. Bisdiazoacetic acid ethyl ester hydrazide is oxidised by nitrous acid to tetrazinedicarboxylic acid ethyl ester azide, $COOC_2H_5.C\overset{N\text{—}N}{<_{N=N}>}C.CO.N_3$, and, similarly, the bishydrazide yields a bisazide. The authors have prepared the neutral hydrazine salt of bisdiazoacetic acid and a series of acid salts of bisdiazoacetic acid; they have thus found that only the monovalent alkali metals and the monovalent diammonium group are capable of forming acid salts.

A Zirconium-mercury Double Salt.—Walter Peters.—Dry pure zirconium tetrachloride reacts with mercury phenyl when they are heated together in a tube which is exhausted by means of a water air-pump. The equation is $ZrCl_4+2Hg(C_6H_5)_2+H_2O=ZrOCl_2.2HgClC_6H_5+2C_6H_6$. The double salt thus obtained is exceedingly soluble in ether. The mercury is evidently not bound directly to the zirconium, for when the double salt is heated *in vacuo* mercury phenylchloride sublimes, and thus the salt is undoubtedly a molecular compound.

Salts of an Acetato-chromium Base.—R. F. Weinland.—By warming chromic acid with glacial acetic acid two crystalline compounds can be obtained. One, a brownish black substance, contains seven molecules of acetic acid and two of chromic acid to three atoms of trivalent chromium, and the other, which is dark green, contains seven molecules of acetic acid and one molecule of chromic acid to three atoms of trivalent chromium. They appear to be chromate acetates of a triacidic acetato-chromium base, $\left[Cr_3{}^{(CH_3CO.O)_6}_{H_2O}\right]^{(OH)_8}$, the first being the bichromate-acetate and the second the chromate-acetate of the same base. Two more chromate-acetates, a chloride-chromate, a nitrate-acetate, and a biacetate may be prepared from this base, as well as a platinum chloride salt. The normal salts of the base have a strong acid reaction in aqueous solution, the basic salts with one hydroxyl group, such as the chloride acetate, have a faint acid reaction. A freshly prepared solution of the chloroplatinate, which contains two hydroxyl groups, is at first neutral, but after a time it becomes feebly acid. All salts of the base with colourless acids are green. The free acetato-chromium base may be prepared in aqueous solution by grinding up an excess of silver oxide in a concentrated solution of the chloride acetate, and filtering after a short time. Thus a dark green alkaline liquid is obtained. This gradually loses its alkaline reaction, and becomes, first, neutral, and finally feebly acid. The acetic acid residue migrates from the cation, and the solution on evaporation gives some silver acetate and biacetate. The acetato base is quite stable in acid solution, but gradually decomposes in the free state. An iron base corresponding to this chromium base is contained in a solution of ferric hydroxide in glacial acetic acid. By the addition of chromic acid to such a solution red crystalline bichromatacetate of formula $\left[Fe_3(CH_3CO.O)_6\right]^{Cr_2O_7.H_2O}_{(CH_3CO.O)}$ is obtained. Similar aluminium compounds also appear to exist. By heating chromic acid with glacial acetic acid and concentrated sulphuric acid, a compound is obtained in the form of a very hygroscopic green powder consisting of trivalent chromium, acetic acid, and sulphuric acid.

Action of Cold Caustic Soda Solution on Cellulose.—W. Vieweg.—When cold caustic soda solution acts on cellulose within concentration limits of 11 to 24 per cent, caustic soda is taken up by the cellulose in the proportions corresponding to the formula $C_{12}H_{19}O_{10}.Na$. The curve representing the absorption of NaOH from caustic soda solution by cellulose has a kink at this stage. If the caustic soda solution is saturated with sodium chloride when the concentration is low, this kink appears at a lower concentration the higher the degree of mercerisation and the greater the amount of common salt in the liquid. Before the kink the curve is irregular, which points to a chemical reaction consisting of an increase in the degree of mercerisation. After the kink the curve becomes a horizontal straight line. The addition of sodium chloride to the alkali up to the saturation point leads to a chemical and physical reaction (adsorption) between caustic soda solution and cellulose.

NOTES AND QUERIES.

Blue Ink.—I was recently shown an engraving printed in blue ink on white paper. Half of this engraving had been soaked in some chemical which had changed the blue ink to a bright green, without in any way affecting the white paper on which it was printed. Green printer's ink can easily be changed to blue by soaking it in oxalic acid, but I have as yet been unable to find out how blue can be changed to green. If any reader is able to give me the information I should be greatly obliged.—F. Baker.

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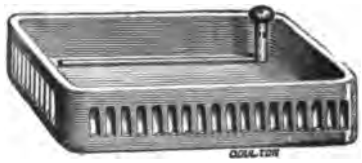

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AND

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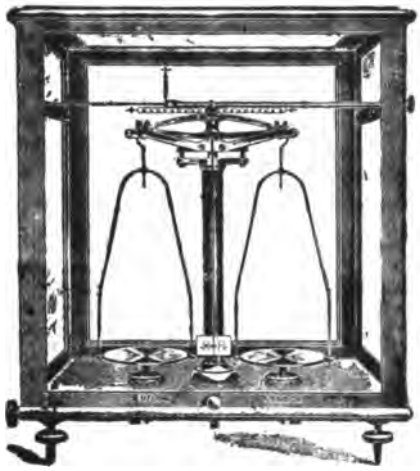

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THE PRESENT STATE OF OUR KNOWLEDGE OF RADIO-ACTIVITY.*

By A. DEBIERNE.

Part I.—*The Discovery of the Radio-active Substances.*

At the present time radio-activity comprises a great number of facts and theories; its development has been extremely rapid, and its importance is quite comparable with that of much older sciences, such as physical chemistry. To show the magnitude of the subject I need only mention that a course of radio-activity, lasting the whole year, is held at the Sorbonne, the professor being Mdme. Curie.

H. Becquerel was the first to observe the fundamental phenomena in the compounds of uranium, and the results of his first experiments show at once what is meant by a radio-active substance.

The salts of uranium possess the property of spontaneously emitting a radiation which is capable of acting on a photographic plate, and of making gases conductors of electricity; when a radio-active substance emits a sufficiently intense radiation the radiation may cause the visible phosphorescence of certain substances.

Various chemical actions may give rise to the phenomena of phosphorescence and to conduction by gases. For instance, the oxidation of phosphorus causes the emission of light, and the surrounding gases become conductors of electricity; the same thing happens with sulphate of quinine, when it is subjected to a rapid change of temperature. But in these cases an essential characteristic of radio-active phenomena is lacking; the production of the phenomena is not spontaneous, but it is caused by external actions which may easily be determined.

Mdme. Curie, by her first experiments, gave a rigorous demonstration of another fundamental property of radio-active substances, namely the atomic nature of the property of radio-activity. This spontaneous emission of rays is an essential characteristic of the uranium atom; it is not influenced by the state of combination, and is maintained unaltered in all the compounds of uranium.

This is a very rare phenomenon, and of all the numerous properties of matter, mass is the only one which persists through transformations of a chemical nature, and which also manifests itself spontaneously.

Some time after Becquerel's discovery, Mdme. Curie and Schmidt showed that the compounds of thorium give rise to the same phenomena as the compounds of uranium.

I.

Exact measurements of the intensity of the radiation emitted by radio-active substances are indispensable in all researches in radio-activity; they are generally performed by determining the conductivity of gases which have been exposed to the rays. The photographic action and the phenomena of phosphorescence cannot be used for measurements, but may be very useful for qualitative observations. I will now shortly describe the different forms of apparatus used in measuring radio-activity.

It has been shown that the conductivity of gases is due to the presence of electrically charged particles which are called ions. The rays which cause the conductivity produce positive and negative ions in the gas; these move in an electric field, and deposit their charges on electrified substances.

The current of electricity produced by this movement of the ions may be measured either with a gold-leaf electroscope or else with a quadrant electrometer. The simplest way is to determine the rate of movement of the gold-leaf of the electroscope, or of the needle of the electrometer. In the case of the electroscope the electric field is generally produced between a charged conductor supporting the gold-leaf and a plate upon which the active substance is placed, and which is connected with the case of the instrument. The rate of movement may be measured by observing the gold-leaf with a little microscope provided with a micrometer, and by determining the time taken to move from one fixed division to another.

With an electrometer a specially constructed condenser is generally employed. It consists of two plates; on one of them, which is raised to a high potential, the substance is placed; the other is connected with one pair of the electrometer quadrants. The needle of the electrometer is also raised to a high potential, and the rate of displacement of the luminous spot on a divided scale is determined.

Measurements with the electroscope are very simple to take, and are capable of great sensitiveness; frequent calibrations are necessary, but they may be performed very quickly with a substance of constant activity, such as uranium, and a great deal of valuable work has been done with this instrument.

Nevertheless, the method which gives the most reliable results is that which was employed by Pierre Curie and Mdme. Curie in their researches, and which is called the quartz piezo-electric method. In this method the phenomenon discovered some time ago by Pierre and J. Curie, and called piezo-electricity, is used. The phenomenon is as follows:—When certain crystals are subjected to mechanical deformations electric charges are produced on certain faces. Thus, when a tensile force is applied to a sheet of quartz cut in a certain manner with regard to the axes, charges of electricity equal in magnitude and of opposite sign are produced on two faces. The quantity of electricity depends upon the dimensions of the sheet, and is exactly proportional to the weight to which the tensile force is due. In the quartz piezo-electric method these charges are used to neutralise the charges brought by the gaseous ions to the plate of a condenser; thus, for example, the time is observed in which the charge developed on the quartz plate by the tension of a given weight is exactly neutralised by the electric charges brought by the ions. In this method the measurements are independent of the arrangement of the apparatus, and the absolute value of the currents can easily be calculated.

II.

Besides the elements uranium and thorium, which are the only ones of the chemical elements previously known to show marked radio-activity, other substances exist with an infinitely greater radio-activity.

The discovery of the most important of these new substances is due to Pierre Curie and Mdme. Curie, who at the same time provided a new method of searching for chemical elements. The rapid development of radio-activity is the outcome of this new method of research and of the discovery of radium.

This last discovery is one of the most remarkable achievements effected in physical science, both as regards the logical development of the researches and the manipulative difficulties which have been overcome. It recalls the discovery of spectral analysis by Kirchhoff and Bunsen, and its immediate result—the isolation of the new alkaline metals, cæsium and rubidium. But the difficulties of demonstrating the existence of radium as a new chemical element and of obtaining its salts in the pure state were much greater.

In the first place, Pierre Curie and Mdme. Curie worked out a method of measurement which enabled the new property to be determined exactly; this was the quartz piezo-electrique method, which is still the most accurate for all researches in radio-activity. Then came the experi-

* *Revue Générale des Sciences*, 1908, xix., 691.

ments of Mdme. Curie, which proved indisputably the atomic nature of the new property, and showed that, of the elements then known, only uranium and thorium are radio-active to an appreciable extent. Then minerals were examined; only those which contain uranium or thorium were found to be appreciably radio-active. But here an important anomaly was observed; the radio-activity of the minerals was sensibly greater than that which would be due to a certain proportion of uranium or of thorium. Hence, in spite of the novelty of the phenomenon, the following conclusion was drawn, viz., that the minerals must contain new chemical elements more strongly radio-active than uranium and thorium. Experiments to separate them were immediately begun.

A small quantity of pitchblende was subjected to chemical treatment by the two physicists, who had no equipment for this kind of work, but hoped that their incursion into the domains of chemistry would be brief, and that they would soon be able to return to the purely physical study of the phenomenon. A first confirmation was soon obtained; substances more radio-active than uranium and thorium can be separated. The existence of new chemical elements in these substances was immediately affirmed; one received the name polonium, the other was called radium. The first hypothetical element is allied to the metals which are precipitated by sulphuretted hydrogen, the second to the metals of the alkaline earths. The results of the separations were always substances which were more and more active, of course in diminishing quantities, and it was soon evident that a larger amount of material would have to be treated. Several kilogrms. were used, and then the rudimentary equipment which was at the disposal of the investigators made the chemical operations very tedious: in spite of this new attempt the same result was obtained; that is to say, substances of increasing activity were prepared, but the activity did not tend towards a limit, and when all the activity had been accumulated in some centigrms. of substance, the latter always appeared to contain a very large proportion of inactive elements. However, during these experiments a confirmation of the existence of one of the new elements gave much encouragement to the investigators to continue their researches. The material containing the hypothetical element radium was examined spectroscopically by Demarçay, who detected a new line, at first very faint, beside those of barium, of which the substance was principally composed; as the activity was concentrated the line became more distinct. The exceeding faintness of the new line showed that the only hope of obtaining pure salt lay in treating several tons of material, and this enormous piece of work was at once begun. The mineral being too expensive, waste products which might contain the new element were obtained from a manufactory; the first treatment was carried out in a factory; the last concentrations, which comprised several hundred fractional crystallisations, were performed in the laboratory, still with the same rudimentary equipment. And after several years of incessant work a sufficient quantity of pure salt (about 1 decigrm.) was obtained. It gave an entirely new spectrum, and the atomic weight of the new element could be determined. According to Mdme. Curie's latest determinations the value is 226·5.

Radium is the only new radio-active substance which has been obtained in a state of purity; it must be mentioned that it is also the only substance the isolation of which has been seriously attempted.

The chief very strongly radio-active substances which have been obtained besides radium and polonium are actinium, which I discovered in pitchblende, radio-thorium, which was discovered by Hahn in thorianite, and ionium, which has quite recently been described by Rutherford and Boltwood. We shall see presently that radio-thorium is closely related to thorium and polonium to radium.

The chemical differences between the various substances are as follows:—Radium, which is the only one that has been obtained as a pure salt, has well characterised chemical properties, which show clearly that it is the last member of the series of the alkaline earths. Its sulphate is still more insoluble in water than barium sulphate; its soluble salts (chloride, bromide, nitrate) are isomorphous with those of barium; the complete separation of the two elements has been effected by fractional crystallisation.

The properties of the metal would be of great interest, but its preparation appears to be as difficult as that of metallic barium, and it has not yet been effected. The operation would involve serious risks of loss of material, and it can readily be understood that the investigators hesitate to undertake it.

In many reactions—precipitation by sulphuretted hydrogen, formation of basic salts—polonium is carried down with bismuth; it may, however, be precipitated by stannous chloride with mercury and tellurium; it may also be partially deposited on plates of silver, bismuth, and even platinum. It has been shown that its activity gradually diminishes.

Actinium has been obtained mixed with the earths which are precipitated by oxalic acid in acid solution, and which do not dissolve in hydrofluoric acid; thus it accompanies thorium and the rare earths. If a fractionation is carried out by the method of the double nitrates of the magnesium series, it passes into the more soluble portions of the ceric earths with neodymium and samarium.

Finally, with regard to radio-thorium but little is known as to its chemical properties; it is only known that it is generally carried down with iron.

As a matter of fact, except in the case of radium, the salts of which have been obtained in a pure state, no definite information can be given regarding the chemical properties of these substances until they have been completely separated from the foreign matter which accompanies them.

In spite of the intense radio-activity which some of them exhibit, they are still in a very diluted state, and the chemical separations which may be observed must be regarded only as the carrying down of them by certain known elements. This has not been understood by some experimenters, who, basing their opinion upon slight chemical differences, have reported the existence of new substances, which have afterwards been recognised as identical with substances already known.

Thus a substance identical with polonium has for some time been called radio-tellurium; another, which is identical with actinium, has received the name emanium. To avoid such confusion we must not rely only upon chemical concentration processes, but the special radio-active properties of each substance must be examined. We shall see that it is very easy to distinguish the different substances from one another, using their radio-active properties only.

(To be continued).

THE

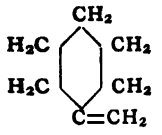
STUDY OF HYDRO-AROMATIC SUBSTANCES.*

(Concluded from p. 228).

Recent Work on Hydro-Aromatic Substances.

Hydrocarbons. — Methylenecyclohexane (I.) has been submitted to detailed investigation by Faworsky and Borgmann (*Ber.*, 1907, xl., 4863), who conclude that the hydrocarbons of this supposed constitution, previously described by Sabatier, Wallach, and Zelinsky and Gutt, are in reality mixtures of methylenecylohexane and 1 : methyl-Δ^1-tetrahydrobenzene (II.). For the preparation of the pure hydrocarbon cyclohexylcarbinol, obtained by the Grignard reaction from bromohexahydrobenzene, was converted into the corresponding iodide, and this latter body heated with alcoholic potash.

* Report of the Committee, consisting of Dr. E. Divers (Chairman), Prof. A. W. Crossley (Secretary), Prof. W. H. Perkin, Dr. M. O. Forster, and Dr. H. R. Le Sueur. Read before the British Association (Section B), Dublin Meeting, 1908.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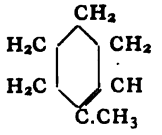
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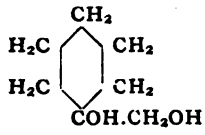
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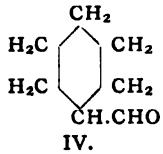
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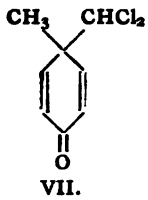
Methylenecyclohexane absorbs two atoms of bromine, and the resulting dibromide gives, on warming with 10 per cent potassium carbonate solution, the glycol having Formula III. When treated with very dilute sulphuric acid this glycol gives an aldehyde (IV.), which can also be obtained directly from the dibromide by treatment with lead oxide and water. These facts conclusively prove the constitution of the dibromide and of the hydrocarbon.

Methylenecyclohexane is unaltered on boiling with quinoline, but is converted by a mixture of quinoline and quinoline-hydriodide into the isomeric methyltetrahydrobenzene (II.). This fact would account for the hydrocarbon obtained by Zelinsky and Gutt from the iodide of cyclohexylcarbinol by heating with quinoline, being a mixture of the two isomerides.

Auwers (*Annalen*, 1907, ccclii., 219, 273, 288) has shown that the alcohols V. and VI., prepared from the ketone VII. by Grignard's reaction, lose water to give unstable intermediate products, which readily pass into benzene derivatives. These reactions—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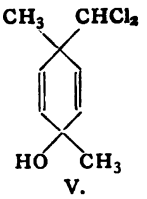
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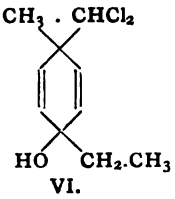
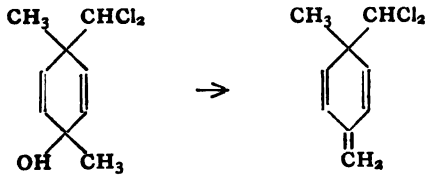
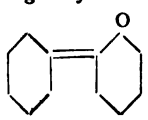

VI.

have been very fully investigated, and are described in detail in this lengthy memoir. The hydroaromatic alcohols V. and VI. and their homologues are not very stable substances, and many, if not all, exist in two modifications, probably due to *cis* and *trans*-isomerism. The above-mentioned intermediate products are considered to be alkylidenedihydrobenzenes—



the general chemical and physical properties of which substances are discussed, compared with other closely related compounds, and shown to support the above conclusions as to their constitution.

Ketohexahydrobenzene is acted on by dry hydrogen chloride (Wallach, *Ber.*, 1907, xl., 70) to give a substance having the formula $C_{12}H_{19}OCl$, which loses the elements of hydrogen chloride to give cyclohexene-2-cyclohexanone.



When this dicyclic ketone is reduced it yields the corresponding saturated alcohol, and this, by treatment with hydrogen iodide, the fully hydrogenised dicyclohexyl $C_6H_{11}.C_6H_{11}$.

Alcohols. — Aromatic quinones give (Sabatier and Mailhe, *Comptes Rendus*, 1908, cxlvi., 57), when treated with hydrogen in presence of reduced nickel, a variety of products, the nature of which depends largely on the reaction temperature ; if moderately low the dihydric alcohols of the hexahydrobenzene series are formed.

Monohydric alcohols may be obtained (Hell and Schall, *Ber.*, 1907, xl., 4162) by the action of various alkyl magnesium iodides on ethyl hexahydrobenzoate. The latter is readily prepared by treating cyclohexylmagnesium iodide with carbon dioxide.

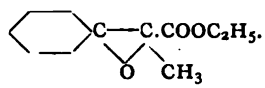
Ketones.—The action of light on ketohexahydrobenzene and the three isomeric ketomethylhexahydrobenzenes has been studied by Ciamician and Silber (*Ber.*, 1908, xli., 1071), who find that the ring is broken with production of a fatty acid containing the same number of carbon atoms together with the corresponding unsaturated aldehyde.

Not only ketohexahydrobenzene itself (*B.A. Reports*, 1907, 110), but also a number of other simple ring ketones, such as ketomethyl- and ketomethylisopropylhexahydrobenzene, are found to be capable of reacting in the enolic form (Mannich and Hänen, *Ber.*, 1908, xli., 564).

The influence of a very reactive agent, for example acetic anhydride, is necessary to bring about the change, and the resulting acetates are converted on saponification into the original ketonss.

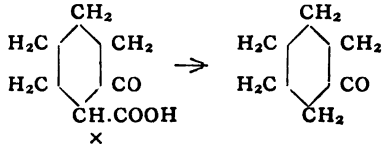
Homologues of ketohexahydrobenzene may be prepared by the slow distillation of the anhydrides of substituted pimelic acids (Blanc, *Comptes Rendus*, 1907, cxliv., 1356).

Ketohexahydrobenzene and the three isomeric ketomethylhexahydrobenzenes condense with ethyl *a*-chloropropionate (Darzens, *Comptes Rendus*, 1907, cxliv.) in presence of sodium ethoxide, to the corresponding glycidic esters, the product in the first case having the formula—

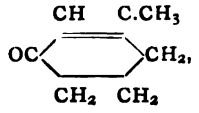


On saponification these esters give free acids, which decompose on distillation in a vacuum, giving hexahydroacetophenone or its homologues. This latter ketone is, however, best prepared (Hell and Schall, *Ber.*, 1907, xl., 4162) by treating the magnesium compound of cyclohexyliodide with acetaldehyde, and oxidising the resulting methylcyclohexylcarbinol $C_6H_{11}.CHOH.CH_3$ with chromic acid.

In the preparation of ketones by elimination of carbon dioxide from ketocarboxylic acids, Kötz (*Annalen*, 1907, cccIvii., 192) finds that the reaction takes place readily in the case of 1-ketohexahydrobenzene-2-carboxylic acid, but it does not proceed at all with acids and less readily with alkalis, as the hydrogen atom marked with a × is replaced by methyl, ethyl, or isopropyl.

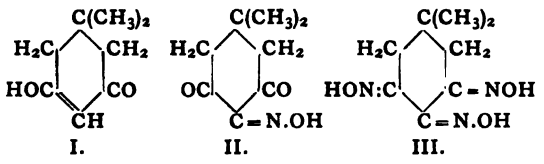


1-Keto-3-methyl-Δ^2-tetrahydrobenzene (Rabe, *Ber.*, 1907, xl., 2482)—

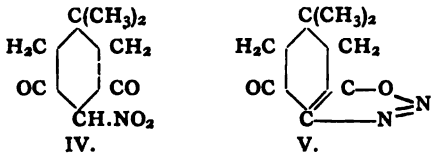


exists in two isomeric forms, which must have the same structure, as on oxidation with potassium permanganate both yield γ-acetobutyric acid. The molecular refraction would lead to the conclusion that both are doubly unsaturated alcohols and not ketonic in nature. Chemically the two forms are identical except in their behaviour towards ferric chloride, when the one gives a pale wine-red colour, whereas the other becomes violet. The present data do not suffice to explain this case of isomerism.

Dimethyldihydroresorcin (I.) reacts towards nitrous acid as a diketone (Haas, *Journ. Chem. Soc.*, 1907, xci., 1433), for the resulting isonitrosodimethyldihydroresorcin (II.) reacts with two molecular proportions of hydroxylamine to give a compound of Formula III. Oxides of nitrogen con-

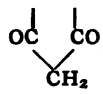


I. II. III.

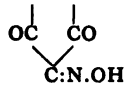


IV. V.

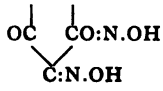
vert dimethyldihydroresorcin into the nitro-derivative (IV.), which substance, on reduction, yields an amine, and this gives with nitrous acid a compound whose constitution may be represented by Formula V. The relation of colour to constitution in these compounds is somewhat striking. Thus, while dimethyldihydroresorcin—



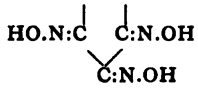
is colourless, isonitrosodimethyldihydroresorcin—



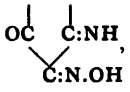
is yellow; its potassium and ferrous salts are deep blue, and its ammonium salt violet: the monoxime—



is colourless, but dissolves in alkalis to a yellow solution, and gives with ferrous sulphate a deep azure blue. The dioxime—

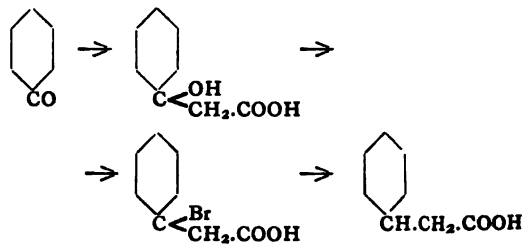


on the other hand, is greyish pink, but gives a yellow solution in caustic alkalis, and produces with ferrous sulphate a violet colour. Replacement of one of the carbonyl oxygen atoms in isonitrosodimethyldihydroresorcin by the imino group,—



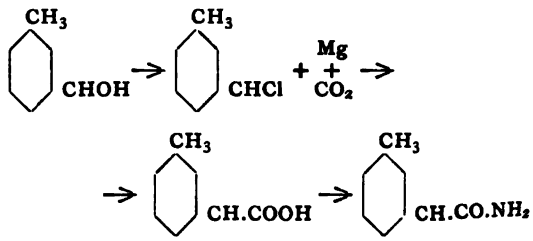
changes the colour from yellow to deep violet; the potassium salt of this compound is magenta, while the ferrous salt is deep blue.

Acids.—A number of carboxylic acids of the hydroaromatic series have been synthesised by Wallach (*Cent. Blatt.*, 1907, ii., 52) by condensing cyclic ketones with ethyl bromoacetate. The hydroxyl group in the resulting esters is replaced by bromine through the agency of hydrogen bromide, and the halogen free acids obtained by treatment with zinc and acetic acid.



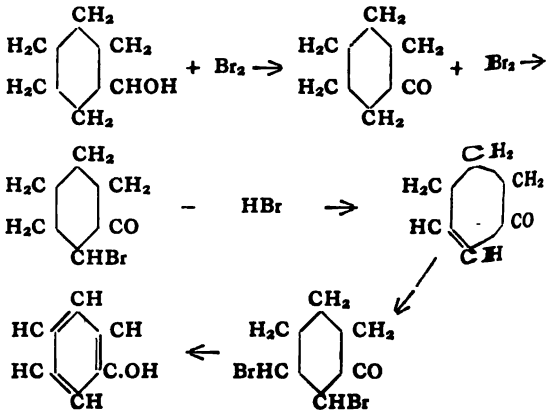
Skita (*Ber.*, 1907, xl., 4167) concludes from a study of ethyl 4-amino-2 : 6 : 6-trimethylhexahydrobenzene-1-carboxylate, prepared from ethyl isophoronecarboxylate, that hydroaromatic amino acids show *cis-trans* isomerism; that they are similar in their chemical behaviour to the aliphatic amino-acids, and also physiologically, in that they possess the property of producing local anæsthesia.

Amines.—Gutt (*Ber.*, 1907, xl., 2061) has prepared hexahydrobenzylamine and the five theoretically possible aminomethylhexahydrobenzenes, which are easily characterised by the melting-points of their benzoyl derivatives. The preparation is realised as follows:—



On treatment with bromine in presence of sodium methoxide the amide gives a urethane, which on distillation with lime yields the amine.

Aromatic from Hydroaromatic Substances.—Kötz and Götz (*Annalen*, 1908, cccl viii., 183) have studied the action of chlorine or bromine on hydroxyhexahydrobenzene and on hexahydrosalicylic acid, and have isolated a number of products intermediate between the fully saturated hydroaromatic bodies and the corresponding aromatic derivatives. An indication of the course of the various reactions may be gleaned from the following scheme:—



Reference may also be made to the following papers, which contain a description of many interesting hydroaromatic substances:—"Hydroaromatic Sulphur Compounds" (Borsche and Lange, *Ber.*, 1907, xl., 2220), "Hydrophthalic Acids" (Abati, *Journ. Chem. Soc.* Abst., 1907, i., 419), "Synthesis of Alcohols of the Hexahydrobenzene Series" (Sabatier and Mailhe, *Ann. Chim. Phys.*, 1907, [8], x., 527), "Derivatives of Phenylcyclohexane" (Kursanow, *Journ. Chem. Soc.* Abst., 1907, i., 599), "Hexahydrobenzaldehyde" (Zelinsky and Gutt, *Ber.*, 1907, xl., 3050, 3055).

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

Part III.—Analysis of the Aluminium and Iron Groups, including Beryllium, Uranium, Vanadium, Titanium, Zirconium, and Thallium.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 229).

Confirmatory Experiments and References (continued).

Action of H_2S *on Molybdate dissolved in excess of* NH_4OH. — A solution containing 6 mgrms. Mo as $(NH_4)_2MoO_4$ and 4 cc. HCl (1·12) in 30 cc. was treated by P. 51: no precipitate formed with NH_4OH, nor with $(NH_4)_2S$. To the nearly colourless filtrate were added 5 cc. NH_4OH (0·90), and H_2S was led through the solution in a test-tube for twenty minutes: after three minutes the solution was dark yellow, after five minutes it had a reddish colour, after ten minutes it was brilliant red, and the colour did not change in the next ten minutes. The colour was not the same as in the case of vanadium, but might be mistaken for it if a comparative test were not made. 50 mgrms. Mo as $(NH_4)_2MoO_4$ and 3 grms. NH_4Cl were dissolved in 100 cc. water, 5 cc. NH_4OH (0·90) were added, and the solution was saturated with H_2S: the colours obtained were the same as in the preceding experiment. The last experiment was repeated with 1 mgrm. Mo: the solution finally became deep orange in colour.

Action of Acids on Sulphomolybdate.—50 mgrms. Mo as $(NH_4)_2MoO_4$, 3 grms. NH_4Cl, and 5 cc. NH_4OH (0·90) were dissolved in 100 cc. water, and saturated with H_2S. The red solution was filtered and then made distinctly acid with acetic acid: the colour remained nearly the same; on filtering, a very small black precipitate was obtained which contained less than 1 mgrm. Mo. The solution was allowed to stand for one hour: it remained clear. After three hours a small precipitate had separated which contained only 2 or 3 mgrms. Mo. The mixture was then boiled for five minutes and filtered: about two-thirds of the molybdenum precipitated as MoS_3, and the filtrate was still deep orange. The filtrate was evaporated to a small volume, and 10 cc. HCl (1·20) added: a large black precipitate of MoS_3 separated, but the solution was found to still contain 1 or 2 mgrms. Mo.

The foregoing experiment was repeated, except that HCl was added to the cold solution: a large black precipitate separated at once. The filtrate was evaporated to 30 cc. and tested for Mo by *P. 43*e: none was found. The experiment was repeated except that the mixture was boiled after adding HCl: the result was the same, not more than a trace of molybdenum being found in the filtrate.

P. 52, N. 3: *The Formation of Sulphate on dissolving* NiS *in Acids.*—In two experiments 300 mgrms. Ni as freshly precipitated NiS were treated by P. 52, the sulphur was filtered off, and $BaCl_2$ was added to the filtrate: small precipitates of $BaSO_4$ separated, each of which was estimated to contain 5 to 10 mgrms. Ba. In another series of experiments the NiS was first treated in the cold for several minutes with mixtures of 5 cc. HNO_3 (1·20) with 20 to 20 cc. water, and the residue was dissolved by evaporating the solution to concentrate the HNO_3, and then adding a few drops HCl (1·20): precipitates of $BaSO_4$ resulted, estimated to contain 20 to 50 mgrms. Ba. In each of these experiments, and especially when the precipitate was first treated with HNO_3, considerable sulphur separated, and the NiS enclosed in it did not dissolve readily.

P. 52, N. 4: *Action of* HCl *on Titanium and Zirconium Hydroxides.*—Acid solutions containing 100 mgrms. Ti and 100 mgrms. Zr as chlorides in volumes of about 100 cc. were treated in the cold with NH_4OH; the precipitates were filtered off and treated with 20 cc. cold HCl (1·12): they dissolved completely in a minute or two. The experiments were repeated, except that the solutions were heated to boiling, and the NH_4OH was added to the hot solutions: on pouring 20 cc. HCl (1·12) repeatedly through the filters containing the hydroxide precipitates, almost all of the $TiO(OH)_2$ dissolved, but only a small portion of the $ZrO(OH)_2$ (8 to 10 mgrms. Zr). On pouring a 20 cc. portion of hot HCl (1·12) repeatedly through the filters, the remainder of the titanium, but only about half the zirconium, dissolved. The remainder of the $ZrO(OH)_2$ was boiled in a casserole with HCl (1·12) for several minutes: it dissolved completely.

P. 52, N. 5: *Complete Precipitation of Titanium and Zirconium in the Sodium Peroxide Procedure.*—1 mgrm. Ti as $TiCl_4$ was treated with NaOH and Na_2O_2 by P. 52, the mixture being boiled for about four minutes after the Na_2O_2 was added: a white precipitate separated. The filtrate was made acid with HCl; one-half of it was tested for titanium by adding H_2O_2: no colour appeared, showing that the titanium had been completely precipitated. The other half of the filtrate was tested for H_2O_2 by adding excess of $TiCl_4$: no colour appeared, showing that the Na_2O_2 had been completely decomposed. The experiment was repeated with 500 mgrms. Ti: the filtrate contained 1 or 2 mgrms. Ti. The precipitate was treated with HCl (1·12): nearly all dissolved in the cold, and the remainder on warming. The solution was reddish yellow, showing that the precipitate contained some TiO_3. The HCl solution obtained in the last experiment (containing nearly 500 mgrms. Ti) was treated with NaOH and Na_2O_2 by P. 52; the mixture containing Na_2O_2 was boiled for less than one minute: the filtrate contained at least 5 mgrms. Ti. These experiments show that the amount of titanium that remains in the filtrate may be greatly lessened by long continued boiling.

1 mgrm. Zr as $ZrCl_4$ was treated with NaOH and Na_2O_2 by P. 52: a distinct precipitate separated. The experiment was repeated with 100 mgrms. Zr; the large precipitate was filtered off, and the filtrate was tested for zirconium by acidifying with HNO_3, evaporating almost to dryness, and adding Na_2HPO_4 solution: only a trifling precipitate separated, thus proving that the precipitation of the zirconium had been complete.

Action of NaOH *on Uranyl Salts.*—A solution containing 5 mgrms. U as UO_2Cl_2 and a little HCl in 15 cc. was neutralised with 10 per cent NaOH solution, and an excess of 2 or 3 cc. was added: a small yellowish precipitate separated. This was filtered off and the filtrate was tested for uranium by acidifying and adding NH_4OH: a small precipitate separated, which was estimated to contain 2 or 3 mgrms. U. The experiment was repeated, except that the mixture containing NaOH was boiled for a minute or two: nearly all of the precipitate which had formed in the cold was re-dissolved. Therefore the uranium is not completely precipitated by a small excess of NaOH. 50 mgrms. U as $UO_2(NO_3)_2$ were added to 30 cc. 10 per cent NaOH solution, and the mixture was heated to boiling, cooled, and poured through a hardened filter; half of the filtrate was tested for uranium as above: only a small precipitate of $(NH_4)_2U_2O_7$ separated, showing

* From the *Journal of the American Chemical Society*, xxx., No. 4.

that the uranium had been nearly completely precipitated by the alkali.

Composition of Sodium Aluminate, Zincate, Beryllate, and Chromite.—For the evidence that sodium aluminate in solution has the formula $Na+AlO_2-$, see Noyes and Whitney (*Zeit. Phys. Chem.*, 1894, xv., 694); Hantzsch (*Zeit. Anorg. Chem.*, 1902, xxx., 296). Hantzsch has shown by means of conductivity measurements that a solution of the empirical composition Na_3AlO_3 contains mainly NaOH and $NaAlO_2$, and some colloidal $Al(OH)_3$.

Hantzsch (*Zeit. Anorg. Chem.*, 1902, xxx., 298, 303) concluded from conductivity measurements that a 1/200 molal solution of sodium zincate, even in the presence of a sevenfold excess of NaOH, is almost completely hydrolysed into NaOH and $Zn(OH)_2$, and that the latter is present as a colloid. Fisher and Herz (*Zeit. Anorg. Chem.*, 1902, xxxi., 355) confirmed this by dialysis experiments. Kunchert (*Zeit. Anorg. Chem.*, 1904, xli., 343), working with a larger excess of alkali, and using Bodländer's electromotive force method, proved, however, that the solutions contained chiefly $ZnO_2=$ and some $HZnO_2-$. Forster (*Zeit. Elektrochem.*, 1899, vi., 301) has prepared solid $NaHZnO_2$.

Hantzsch (*loc. cit.*) concludes that H_2BeO_2 is a very weak acid, weaker than $HAlO_2$, but much stronger than H_2ZnO_2.

From dialysis and conductivity experiments Fisher and Herz (*loc. cit.*) conclude that in alkaline chromite solutions chromium is present almost solely as colloidal $Cr(OH)_3$.

Behaviour of Cobalt Hydroxide towards NaOH.—See Donath (*Zeit. Anal. Chem.*, 1901, xl., 137).—An acid solution containing 100 mgrms. Co as nitrate in 30 cc. was neutralised with 10 per cent NaOH and an excess of 15 cc. added; 2 grms. Na_2O_2 were added a little at a time to the cold solution, the mixture was boiled for two or three minutes, cooled, and filtered: the filtrate had a deep blue colour. It was acidified, made alkaline with NH_4OH, and H_2S was passed into it: a precipitate separated which was estimated to contain 5 to 8 mgrms. Co. The experiment was repeated, except that no excess of NaOH was added: the filtrate was colourless and no cobalt was found in it. This last experiment was repeated except that 4·5 grms. Na_2O_2 were used instead of 2 grms.: again the filtrate contained no cobalt. Therefore the blue (soluble) cobalt compound is formed only by the action of concentrated alkali on a *cobaltous* salt, and the cobalt is completely precipitated if it is first oxidised to the cobaltic state by Na_2O_2 in a weakly alkaline solution.

The first experiment with the large excess of NaOH was repeated with 100 mgrms. Ni instead of Co: no nickel was found in the filtrate.

P. 52, N. 6: *Formation of Peruranates.*—See P. Melikow and L. Pissarjewsky (*Zeit. Phys. Chem.*, 1899, xxviii., 556). A large number of salts are known in the solid state; *e.g.*, $UO_4.2Na_2O_2.8H_2O$; $UO_4.2BaO_2.8H_2O$; $UO_4.2BaO_2.10H_2O$; &c. The salts of the alkali elements are soluble in water, the others insoluble. These salts may be regarded as compounds of peruranic anhydride, UO_4, with peroxides of the other elements. They were prepared by the action of H_2O_2 on solutions of uranyl salts in the presence of the hydroxides of the other elements.

A HNO_3 solution containing 100 mgrms. U as $UO_2(NO_3)_2$ in 25 cc. was treated by P. 52: the solution was yellow when acid, and a yellow precipitate separated on adding NaOH; on adding Na_2O_2 (3 grms.) and boiling, a deep red solution resulted. This solution was acidified with HNO_3: it became yellow. To a portion of it was added a little $TiCl_4$ in HCl solution: an orange-red colour resulted, proving the presence of free H_2O_2. Therefore the uranium is oxidised by Na_2O_2 to a soluble "peruranate," which is stable in the strong alkali, but apparently decomposes readily in the acid solution with formation of H_2O_2 and a uranyl salt. The experiment was repeated, except that only about 0·5 grm. Na_2O_2 was added: a yellow uranium precipitate remained, but the solution contained a large proportion of the uranium.

P. 52, N. 7: *Complete Decomposition of* Na_2O_2 *by Boiling.*—2 grms. Na_2O_2 were added to a neutral solution containing 0·5 grm. NaCl in 30 cc., the mixture was heated to boiling, boiled for one minute, cooled, acidified with HNO_3, and tested with $TiCl_4$ solution for H_2O_2; the solution remained colourless. The experiment was repeated with 100 mgrms. Mn as $MnCl_2$, the MnO_2 being filtered off before the filtrate was made acid: it contained no H_2O_2. The experiment was repeated with 100 mgrms. V as Na_3VO_4: the result was the same. See the preceding paragraph as to the effect of uranium.

Precipitation of Thallium by Sodium Peroxide.—1, 3, and 20 mgrms. Tl as $Tl(NO_3)_3$ were treated in separate experiments by P. 52: brown precipitates separated in each case on adding NaOH, and remained after the treatment with Na_2O_2. The solutions, which were filtered off almost immediately after diluting, were found to contain about 0·5, 1, and 2 to 3 mgrms. respectively. 1 mgrm. Tl as Tl_2SO_4 was treated by P. 52: no precipitate separated on adding NaOH; there was a slight coloration on adding Na_2O_2, but no precipitate remained upon boiling; when, however, the solution was set aside to cool, a good precipitate separated in ten or fifteen minutes, and the filtrate was found to contain only 0·2 to 0·4 mgrm. Tl. A mixture containing 1 mgrm. Tl as TlCl, 200 mgrms. Fe as $FeCl_2$, 200 mgrms. Mn as $MnCl_2$, and 200 mgrms. Al as $AlCl_3$ was treated by P. 52: the filtrate was evaporated nearly to dryness and tested for thallium by *P. 65*d*: no precipitate of TlI separated.

P. 52, N. 8: *Necessity of Adding* Na_2CO_3 *to Precipitate Barium.*—1 and 2 mgrms. Ba as $BaCl_2$ were treated by P. 52, the final volume being about 30 cc.: on boiling, a precipitate which was proved to contain barium appeared in both experiments, but it was very slight in that with 1 mgrm. These experiments were repeated, except that no Na_2CO_3 was added: no precipitate appeared on boiling.

2 mgrms. Cr and 100 mgrms. Ba as chlorides were treated by P. 52, except that no Na_2CO_3 was added: the filtrate was colourless, and the solution obtained on dissolving the precipitate in HNO_3 was yellow, showing that the chromium had been precipitated as $BaCrO_4$. The experiment was repeated with 1 mgrm. Cr and 100 mgrms. Ba, except that Na_2CO_3 was added: the filtrate was yellow, showing that the addition of Na_2CO_3 causes chromium to pass into the filtrate even when a large amount of barium is present.

Solubility of Zinc Carbonate in NaOH.—50 mgrms. Zn as nitrate were dissolved in 20 cc. water and an equivalent amount of Na_2CO_3 added: a white precipitate separated. 5 cc. more 10 per cent NaOH solution were added: the precipitate dissolved. 5 cc. more NaOH and 10 cc. 10 per cent Na_2CO_3 solution were added, and the mixture heated to boiling: no precipitate separated. More Na_2CO_3 was added: a precipitate finally formed.

P. 52, N. 9: *Solubility of the Phosphates of Aluminium and Zinc in* NaOH.—100 mgrms. Zn and 100 mgrms. Al as phosphates were dissolved in separate experiments in 25 cc. water and a very little HNO_3 (1·20); 10 per cent NaOH was added slowly: precipitates separated but dissolved when an excess of about 4 cc. had been added.

Partial Decomposition of Phosphates on Treating with Sodium Hydroxide.—50 mgrms. Mg, 50 mgrms. Ca, and 20 mgrms. (ferric) Fe, all as freshly precipitated phosphates, were boiled separately with about 30 cc. 4 per cent NaOH solution for three or four minutes in covered casseroles; the mixtures were filtered, the precipitates being washed with dilute NaOH. The filtrates were made strongly acid with HNO_3, and the precipitates were dissolved in HNO_3. Each solution was evaporated to a small volume and treated with several cc. ammonium molybdate solution. The amount of phosphate in each solution was estimated by the amount of yellow precipitate

that had separated out after several hours: in the case of magnesium only a very small proportion of the phosphate remained in the precipitate; in the case of the iron about one-fifth remained in the precipitate; and in the case of calcium the phosphate was about equally divided between precipitate and filtrate.

Behaviour of Calcium Borate and Oxalate towards Sodium Hydroxide.—A mixture containing 100 mgrms. Ca as $CaCl_2$ and 80 mgrms. BO_2 as H_3BO_3 was treated by P. 52: the precipitate was tested for borate by adding to it in a casserole H_2SO_4 (1·84) and alcohol, igniting the alcohol and stirring vigorously: no green colour appeared. The filtrate was acidified with H_2SO_4, evaporated, and tested in the same way: a large amount of borate was found. A trial experiment showed that 5 mgrms. BO_2 could easily be detected in this way.

A mixture containing 500 mgrms. Fe as $FeCl_3$, 500 mgrms. C_2O_4 as oxalic acid, and 100 mgrms. Ca as $CaCl_2$ was treated by P. 52; the filtrate was analysed for oxalate by acidifying, adding NH_4OH and $CaCl_2$: a very large precipitate of calcium oxalate separated. The experiment was repeated with 300 mgrms. Ca and 500 mgrms. C_2O_4 (but no iron): the result was the same. The precipitate was tested for oxalate by dissolving it in HCl and adding NH_4OH: a small precipitate separated, showing that a large proportion of the oxalate had passed into the filtrate.

Decomposition of Oxalic Acid by Acids.—10 and 100 mgrms. oxalic acid were treated separately by P. 61, about 1 grm. $KClO_3$ being added to the concentrated HNO_3 solution. The solutions were finally evaporated to almost 5 cc., diluted to 30 cc., neutralised with NH_4OH, and tested for oxalate by adding 10 cc. of 10 per cent $Ca(NO_3)_2$ solution: no precipitate separated in either experiment. The experiment with 10 mgrms. oxalic acid was repeated, except that no $KClO_3$ was added to the HNO_3 solution: a precipitate containing about 3 mgrms. oxalic acid resulted. 10 mgrms. oxalic acid were added to 15 cc. HCl (1·20), the mixture was evaporated to 2 or 3 cc., diluted and tested for oxalic acid as above: a precipitate of calcium oxalate separated that was estimated to correspond to 3 or 4 mgrms. oxalic acid. The experiment was repeated with aqua regia instead of HCl (1·20): somewhat more oxalic acid was decomposed, but at least 2 mgrms. remained. These experiments prove that the decomposition of oxalic acid is very rapid in a hot mixture of HNO_3 and $HClO_3$, much slower in aqua regia, and still slower in HCl (1·20) or HNO_3 (1·42).

P. 52, N. 10: *Colour of an Alkaline Solution of Sodium Chromate and Peruranate.*—1 mgrm. Cr as $KCrSO_4$ was treated by P. 52, the final volume being about 30 cc.: the yellow colour of the solution was easily seen.

1 mgrm. U as $UO_2(NO_3)_2$ was treated by P. 52, the final volume being about 15 cc.: the solution was distinctly yellow. The experiment was repeated with 5 mgrms. U; the solution had a reddish tinge. It was diluted to 30 cc.: the reddish colour changed to yellow.

(To be continued).

Lectures on Forensic Chemistry.—Mr. W. Jago, F.I.C., of Lincoln's Inn, Barrister-at-Law, will deliver a course of four public lectures on Forensic Chemistry at University College. The first lecture will be given on Friday, November 13th, at 5.30, when the chair will be taken by Mr. A. J. Walter, K.C. The Lectures are open to the public without fee or ticket.

Honours.—The Royal University of Ireland have conferred the degree of D.Sc. *honoris causa* on Prof. Alfred Senier, of Queen's College, Galway, in recognition of his services as a teacher of chemistry in Ireland, and of his researches in organic chemistry, notably his work on acridines.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

The following complete the abstracts of Papers received during the vacation, and published or passed for publication in the *Transactions*:—

184. "*The Interaction of the Metals of the Aluminium Group and Organic Halogen Derivatives.*" By James Frederick Spencer and Marion L. Wallace.

Aluminium, indium, and thallium react directly with both aromatic and aliphatic halogen derivatives on heating. In the case of the chloro-derivatives and the lower aliphatic compounds (up to butane), heating in a sealed tube was necessary to bring about the reaction. The formation of an intermediate aluminium organic halide, similar to the Grignard compounds, is the primary result of this interaction, and this compound is decomposed on the addition of water with the production of the parent hydrocarbon and the evolution of much heat. In some cases, the evolution of heat does not take place, and in these cases the parent hydrocarbon is present in the mixture before the addition of water. In all such cases the reactions are extremely slow, it being necessary to heat the substances for twenty-four to thirty hours before the reaction is complete; so that in all probability the intermediate compound is formed, and then decomposed by the atmospheric moisture. The indium and thallium reactions, of which only few have been studied, proceed very slowly, as, indeed, do also some of the aluminium reactions, but certain of the latter take place with extreme violence. The yield of hydrocarbons varies from 25 to 75 per cent of the theoretical. In a few cases, the reaction proceeds along a second line, namely, the formation of a hydrocarbon or derivative containing double the number of carbon atoms as the original substance. The best examples of this are the formation of dimethyl succinate from methyl bromoacetate, and the formation of benzidine from bromoaniline by the action of aluminium. In no case could a reaction between these metals and the halide derivatives be brought about in ethereal solution.

185. "*The Relation between Unsaturation and Optical Activity.* Part IV. *The Relative Influence of Bi-, Quadri-, and Sexavalent Sulphur on Rotatory Power.*" By Thomas Percy Hilditch. (*Trans.*, 1908, 1618).

The optical activity of derivatives of *l*-amyl alcohol and *d*-camphor containing sulphur of all the above degrees of valency has been compared, and it has been found that a bi- or quadri-valent sulphur atom has a larger effect on optical rotatory power than saturated sexavalent sulphur, and, also, that the optical anomaly is enhanced by the presence of a second unsaturated group adjacent to the sulphur residue. The maximum change of rotatory power appears to take place when two identical unsaturated groups occur in conjugation, as in the disulphides.

In addition, the study of a series of active salts of corresponding sulphinic and sulphonic acids shows that the sulphonic acid exerts a greater influence on the rotatory power of the base, and this agrees with the effect of unsaturation observed in other cases, on the assumption that the sulphinic salts contain sexavalent sulphur.

186. "*Oxidations of Hydrocarbons of the Benzene Series.* Part II. *Substances containing a Negative Radicle.*" By Herbert Drake Law and Frederick Mollwo Perkin. (*Trans.*, 1908, 1633).

The authors have investigated the oxidation of the nitrotoluenes with chromyl dichloride. Contrary to Etard's statement that quinones are produced, *p*- and *m*-nitrotoluene give good yields of the corresponding aldehydes. With *o*-nitrotoluene, no aldehyde is formed, but a chlorinated compound of no definite boiling-point. With *o*-, *m*-, and *p*-chlorotoluenes, the corresponding aldehydes are in all cases obtained. Diphenylmethane yields diphenylcarbinol,

and triphenylmethane gives triphenylcarbinol; in both cases in practically theoretical yields. Naphthalene, anthracene, and fluorene were also oxidised, but definite products were not obtained.

187. "*Coprosterol.*" (Part I.). By Charles Dorée and John Addymann Gardner. (*Trans.*, 1908, 1625).

Having shown that cats and dogs when fed on a raw brain diet produce coprosterol in considerable amount, the authors have studied the action of oxidising and isomerising agents on the substance. Coprosterol is readily oxidised by chromic acid to a ketone, *coprostanone*, which crystallises in leaves melting at 62—63°. The *semicarbazone* melts at 192°, and the *oxime* at 69°. Coprostanone forms with phenylhydrazine a crystalline substance melting at 191°, to which a formula has not yet been assigned. When heated with sodium amyl-oxide, coprosterol is converted into *ψ-coprosterol*, melting at 119°, and yielding an *acetate*, m. p. 83°, and a *benzoate*, m. p. 85°, which differ from the corresponding derivatives of coprosterol. On oxidation with chromic acid, however, it yields the same ketone, coprostanone.

188. "*The Spontaneous Crystallisation of Solutions of some Alkali Nitrates.*" By Bernard Mouat Jones. (*Trans.*, 1908, 1739).

The author has determined the temperatures of spontaneous crystallisation of supersaturated solutions of the nitrates of potassium, rubidium, and cæsium of various strengths. The method was that used by Hartley, Jones, and Hutchinson (*Trans.*, 1908, xciii., 825) in investigating sodium sulphate solutions. The effect of mechanical friction was studied by introducing fragments of Jena glass into the tubes before sealing. Crystalline fragments, such as garnet, were not used, in order to avoid possible unequal "inoculating" effects on the three solutions. The temperatures at which supersaturated solutions of the salts in question would crystallise spontaneously when slowly cooled and constantly shaken have been recorded. Temperatures corresponding with tubes containing glass fragments lie on regular curves, namely, the supersolubility curves, above which crystallisation cannot take place in the absence of crystal nuclei. Tubes containing no glass fragments generally needed to be cooled into the labile region before crystallisation would occur. The supersolubility curves for potassium and rubidium nitrates run nearly parallel to the solubility curves, approaching the latter as the concentration decreases. For cæsium nitrate, the solubility and supersolubility curves coincide, the metastable region disappearing.

The three corresponding ice-supersolubility curves have been traced, and also the ice-solubility curves for dilute solutions of rubidium and cæsium nitrates. The spontaneous production of the ice phase seems to be a somewhat less definite phenomenon than the production of the salt phase.

Although the order of molecular solubility is rubidium nitrate, potassium nitrate, cæsium nitrate, the order for the sizes of the metastable ranges follows that of the molecular weights, that for potassium nitrate being greatest, and that for cæsium nitrate least. For example, for a concentration of 0·6 grm.-mol. of salt to 100 grms. of water, the metastable ranges are approximately 4°, 1°, and 0° respectively.

189. "*Syntheses with Phenol Derivatives containing a Mobile Nitro-group.* Part I. *The Interaction of* 2 : 3 : 5-*Trinitro-4-acetylaminophenol and Amines.*" By Raphael Meldola and James Gordon Hay. (*Trans.*, 1908, 1659).

The trinitroacetylaminophenol referred to was described by one of the authors in 1906 (*Trans.*, lxxxix., 1935), and its great reactivity due to the mobility of the 3-nitro group, indicated in that paper. The compound has now been submitted to further investigation, more especially with reference to the formation of iminazoles from amines. In the present paper, the authors consider the mechanism of the formation of iminazoles in connection with the conditions that determine the mobility of the nitro-group on the one hand, and, on the other, the nature of the amine with which the trinitro-compound is brought into combination. A quantitative method for determining the yield of iminazole has been devised, and complete series of determinations have been carried out with the isomeric chloroanilines, nitroanilines, and anisidines. The results bring out very clearly the influence of steric hindrance in the case of ortho-substituted amines, the nitro-group being so completely protective that no combination takes place with *o*-nitroaniline. Other series of isomerides are under investigation, and also the products of condensation of secondary amines; dimethylamine and piperidine having been found to react readily, whilst no combination takes place with methylaniline, benzylaniline, diphenylamine, or carbazole. A number of new iminazoles have been prepared, and are described in the paper.

190. "*The Constituents of the Expressed Oil of Nutmeg.*" By Frederick Belding Power and Arthur Henry Salway. (*Trans.*, 1908, 1653).

Ceylon nutmegs, of good quality, yielded on expression 26·6 per cent of oil, and when completely extracted with ether, 42·9 per cent of oil was obtained. The constants of the expressed oil and of the total fatty acids were as follows :—

	Expressed oil.	Total fatty acids.
Melting-point	48°	49°
Density, 50°/50° ..	0·9399	0·9012
Acid value	11·2	218·3
Saponification value .	174·6	—
Iodine value	57·8	23·1

The composition of the expressed oil or fat was approximately as follows :—Essential oil, 12·5 per cent; trimyristin, 73·0 per cent; oleic acid, as glyceride, 3·0 per cent; linolenic acid, as glyceride, 0·5 per cent; formic, acetic, and cerotic acids, very small amounts; unsaponifiable constituents, 8·5 per cent; resinous material, 2·0 per cent.

The chief unsaponifiable constituent was a new *compound*, $C_{18}H_{22}O_5$, amounting to about 5 per cent of the expressed oil. It is a yellow, transparent, extremely viscid liquid (b. p. 270—280°/15 mm.), and is devoid of any marked physiological action. The remainder of the unsaponifiable material consisted of myristicin, $C_{11}H_{12}O_3$, with a little phytosterol, $C_{20}H_{34}O$ (m. p. 134—135°).

191. "*The Preparation of Disulphides.* Part IV. *Esters of Dithiodiglycollic and Dithiodilactylic Acids.*" By Thomas Slater Price and Douglas Frank Twiss. (*Trans.*, 1908, 1645).

On submitting the mixture obtained by the interaction of ethyl bromoacetate and sodium thiosulphate in aqueous-alcoholic solution to electrolysis in the cathode compartment of a divided cell, diethyl dithiodiglycollate separates as a heavy oil; from methyl bromoacetate the dimethyl ester is obtained. In a similar manner, the diethyl esters of α-dithio- and β-dithio-dilactylic acids were prepared from ethyl α-bromopropionate and ethyl β-iodopropionate respectively. These esters are all fairly mobile liquids, heavier than water, and possessing a peculiar odour which is not very disagreeable, and not at all like that which is usually associated with the liquid mercaptans and disulphides. The boiling-points of these esters are as follows: —*Dimethyl dithiodiglycollate*, 154°/14 mm.; *diethyl dithiodiglycollate*, 164°/14 mm.; *diethyl α-dithiodilactylate*, 159°/14 mm.; *diethyl β-dithiodilactylate*, 194°/17 mm.

For complete identification of these esters, the corresponding acids were prepared from them by hydrolysis with hydrochloric acid. The properties of the acids obtained agreed with those found by previous observers.

192. "*Contributions to the Chemistry of the Cholesterol Group.* Part I. *The Action of Hydrogen Peroxide and of Fused Potassium Hydroxide on Cholesterol.*" By Robert Howson Pickard and Joseph Yates. (*Trans.*, 1908, 1678).

A detailed account of work, of which a preliminary

account has already appeared (*Proc. Chem. Soc.*, xxiv., 121).

193. "*Derivatives of S-Phenylphenazothionium.*" (Part II.). By Samuel Smiles and Thomas Percy Hilditch. (*Trans.*, 1908, 1687).

Further proof of the constitution of these substances is now offered from a general standpoint. Condensation of phenol with di-*p*-nitrodiphenylamine sulphoxide yields the sulphates of two isomeric S-hydroxyphenylphenazothionium bases, which are distinguished by their different solubilities and colours in various media. The reduction products of the dinitro-S-phenetyl and -hydroxyphenyl derivatives have also been prepared; these on oxidation yield blue dye-stuffs similar to thionine. *iso*Dinitrodiphenylamine sulphoxide yields similar derivatives, which are distinguished from those of the di-*p*-nitro-series by greater solubility and absence of fluorescence.

194. "*The Trithionates and Tetrathionates of the Alkali Metals.*" (Part I.). By John Edwin Mackenzie and Hugh Marshall. (*Trans.*, 1908, 1726).

The authors have prepared a number of tetrathionates from persulphates and thiosulphates (compare Marshall, *Journ. Soc. Chem. Ind.*, 1897, xvi., 396), thus, $2SrS_2O_3 + K_2S_2O_8 = K_2S_4O_6 + 2SrSO_4$, and of trithionates by removal of sulphur, thus,—

$$K_2S_4O_6 + K_2SO_3 = K_2S_3O_6 + K_2S_2O_3.$$

The crystallographic details of some of the salts are as follows:—

Potassium trithionate, $K_2S_3O_6$. Rhombic, bipyramidal. $a:b:c = 0{\cdot}7168 : 1 : 0{\cdot}4193$. Refractive indices: $\alpha = 1{\cdot}4925$, $\beta = 1{\cdot}5646$, $\gamma = 1{\cdot}6014$. Calculated axial angle, $2V = 68° 15'$. Density, 2·336. Molecular volume, 115·8.

Rubidium trithionate, $Rb_2S_3O_6$; isomorphous with the above. $a:b:c = 0{\cdot}7058 : 1 : 1{\cdot}04176$. Refractive indices: $\alpha = 1{\cdot}4874$, $\beta = 1{\cdot}5580$, $\gamma = 1{\cdot}5867$. Calculated axial angle, $2V = 62° 23'$. Density, 2·845. Molecular volume, 127·7.

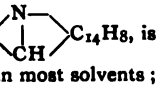
Cæsium trithionate, $Cs_2S_3O_6$, is not isomorphous with the above. Triclinic. Density greater than 3·326.

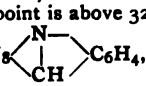
Cæsium trithionate monohydrate, $Cs_2S_3O_6,H_2O$. Triclinic. Density, 3·192. Molecular volume, 149·1.

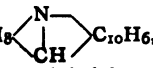
The authors find that rubidium and cæsium can be estimated by conversion into the double sulphate of calcium and the alkali metal.

195. "*The Synthesis of Complex Acridines.*" By Percy Corlett Austin. (*Trans.*, 1908, 1760).

The author has applied the methods employed by Senier and himself in the synthesis of various phenonaphthacridines and dinaphthacridines (*Trans.*, 1906, lxxxix., 1387; 1907, xci., 1233, 1240; 1908, xciii., 63) to the synthesis of acridines derived from phenanthrene and the closely-allied fluorene. A phenophenanthracridine has also been prepared by the condensation of phenanthraquinone and *o*-nitrobenzyl chloride in the presence of stannous chloride. This is an adaptation of Baezner's reaction (*Ber.*, 1904, xxxvii., 3077).

Diphenanthracridine, $C_{14}H_8$  $C_{14}H_8$, is a yellow crystalline substance, insoluble in most solvents; it can be distilled under diminished pressure, and re-crystallised from nitrobenzene. Its melting-point is above 320°.

Phenophenanthracridine, $C_{14}H_8$  C_6H_4, is prepared by treating an alcoholic solution of a mixture of phenanthraquinone and stannous chloride with *o*-nitrobenzyl chloride. After distillation under diminished pressure, it crystallises from benzene in yellow crystals, melting at 204°.

Fluorene- *-naphthacridine*, $C_{13}H_8$  $C_{10}H_6$, is prepared from 2-aminofluorene and α-naphthol by the action of methylene di-iodide. After distillation under diminished pressure and re-crystallisation from toluene, it melts at 259°.

A brown powder is also obtained by the action of methylene di-iodide on 2-aminofluorene, but no method was found for the purification of this substance.

196. "*A Reaction Distinguishing Phosphoprotein from Nucleoprotein, and the Distribution of Phosphoproteins in Tissues.*" By Robert Henry Aders Plimmer and Frederick Hughes Scott. (*Trans.*, 1908, 1699).

In 1906, Plimmer and Bayliss (*Journ. Physiol.*, xxxiii., 439) found that the phosphorus of caseinogen was completely eliminated as phosphoric acid by the action of 1 per cent aqueous sodium hydroxide at 37° in twenty-four hours.

The phosphorus of vitellin, the other well-defined phosphoprotein, is eliminated as phosphoric acid under the same conditions, but the phosphorus of nucleic acid, lecithin, and glycerophosphoric acid, the other phosphorus-containing substances in the tissues, is not separated in this way.

The reaction thus serves as a simple means of distinguishing phosphoproteins from nucleoproteins, and it permits of the determination of the presence of phosphoproteins which are stated to occur in various tissues.

As phosphates are present in all tissues, the reaction cannot be applied directly, but by first extracting the fats and lecithin with alcohol and ether, dissolving out the phosphates from the coagulated protein with water acidified with hydrochloric acid, and then treating it with 1 per cent aqueous sodium hydroxide at 37°, the presence of phosphoprotein can be demonstrated. By this procedure, a determination of the amount of the various kinds of phosphorus compounds in the tissues can also be made.

The pancreas alone of the tissues examined contained phosphoprotein, the amount of phosphorus present in this form being about 3 per cent of the total phosphorus.

Ichthulin, the protein contained in caviar, the eggs of the sturgeon, is described as a phosphoprotein. Its nature has been confirmed by this method, and phosphoproteins have also been found in the eggs of the herring and mullet.

The principal distribution of phosphoproteins is thus in those substances which form the food-stuffs of the embryo bird and fish and the young mammal.

197. "*The Colouring Matters of the Stilbene Group.* Part V. *The Action of Caustic Alkalis on Derivatives of Para-nitrotoluene.*" By Arthur George Green and James Baddiley. (*Trans.*, 1908, 1721).

Derivatives of *p*-nitrotoluene containing as ortho-substituent electro-positive or weakly electro-negative groups (CH_3, OMe, or CO_2H), react least readily with caustic alkalis, giving red intermediate compounds, and on oxidation, derivatives of dinitrodibenzyl. By carrying the combined condensation and oxidation further, the dinitrodibenzyl derivatives are finally converted into dinitrostilbene derivatives, apparently by intermediate formation of nitronitrosostilbenes:—

$$\begin{array}{l} CH_2{\cdot}C_6H_4{\cdot}NO_2 \\ | \\ CH_2{\cdot}C_6H_4{\cdot}NO_2 \end{array} \xrightarrow[\text{Condensation}]{} \begin{array}{l} CH{\cdot}C_6H_4{\cdot}NO \\ \| \\ CH{\cdot}C_6H_4{\cdot}NO_2 \end{array} \xrightarrow[\text{Oxidation}]{} \begin{array}{l} CH{\cdot}C_6H_4{\cdot}NO_2 \\ \| \\ CH{\cdot}C_6H_4{\cdot}NO_2 \end{array}.$$

On the other hand, 2:4-dinitroluene, like other derivatives of *p*-nitrotoluene with a strongly electro-negative ortho-substituent (CN, SO_3Ph, &c.), is far more reactive, giving an excessively unstable blue intermediate product, which is oxidised to tetranitrostilbene without the formation of any dibenzyl compound. The conclusion is drawn that the red intermediate products are nitronitrosodibenzyl derivatives, whilst the blue intermediate compounds are dinitrosostilbene derivatives, both classes of nitroso-compounds existing in an alkaline solution in the form of quinonoid alkali salts.

4 : 4'-*Dinitro*-2 : 2'-*dimethylstilbene*, obtained from *p*-nitro-*o*-xylene, forms yellow needles, which melt at 288—290°. 4 : 4'-*Dinitro*-2 : 2'-*dimethoxystilbene*, prepared from *p*-nitro-*o*-cresol methyl ether, crystallises in orange needles, melting at 268—269°. 4 : 4'-*Dinitrostilbene*-2 : 2'-*dicarboxylic acid*, obtained from nitro-*o*-toluic acid, crystallises from cresol in pale yellow crystals, which melt and decompose at 288—291°. 2 : 4 : 2' : 4'-Tetranitrostilbene is formed by the action of cold alcoholic potash and iodine on 2 : 4-dinitrotoluene. 2 : 4 : 2' : 4'-Tetra-aminostilbene forms pale yellow leaflets, which melt at 183—186° (Echales, *Ber.*, 1904, xxxvii., 3599, gives 191° or below).

198. "*The Solubility of Lime in Water.*" By GERALD TATTERSALL MOODY and LEWIS THOMAS LEYSON.

The widely divergent numbers giving the solubility of lime in water recorded by previous observers are to be ascribed to the use of impure lime, and to faulty methods of determining the solubility. The observed difference in the solubility of lime obtained from different sources is not ascribable to any peculiarity of the lime itself, but is due to the presence of impurities which either affect the solubility of the lime or are capable of neutralising acids, thereby causing an error wherever the lime present in solution is estimated by titration with acid.

An apparatus specially devised for eliminating the errors in method made by previous observers has allowed figures showing the solubility of lime between 2° and 80° to be obtained, and it is found that the curve of solubility between 25° and 80° is practically a straight line.

PHYSICAL SOCIETY.

Ordinary Meeting, October 23rd, 1908.

THE meeting was held at the National Physical Laboratory, Bushy House, Teddington, by kind invitation of the Director. All departments of the Laboratory were thrown open for inspection, and, in addition, a number of special demonstrations had been arranged.

In the Engineering Department Dr. STANTON described a new Fatigue Testing Machine for Metals. For comparison, one of the earliest types of fatigue testing machines—viz., that used by Wöhler—was shown. Here alternations of stress are produced by the rotation of a loaded cantilever. This method is only useful at low speeds.

The new machine is suitable for very great changes in speed, and lends itself to a determination of the effect of rapidity of reversals on the life of the specimen. The test-piece, in the form of a short hollow cylinder, is placed between three rollers, the upper one of which is loaded by a lever and weights. The rollers are supported on friction wheels. The maximum stresses occur on the inside of the ring, the variation being from a maximum tension at the points of contact with the rollers to a maximum compression midway between. There are therefore three cycles of stress for each revolution of the specimen, and no difficulty is found in imposing 2500 cycles per minute at a moderate speed of rotation of the rollers.

Mr. BAIRSTOW showed a Single-lever Testing-machine for producing alternations of direct stress, specially designed with compound knife-edges so that the change from tension to compression can be made by moving the jockey. The extensions of the specimen are observed at intervals on a Marten's Extensometer. The rate of reversals is necessarily low, being only about 100 per hour. The object of the experiments is to determine the elastic range under variation of stress.

In the Electro-technical Department Mr. PATERSON and Mr. RAYNER showed the appliances and arrangements for testing alternating-current instruments. The method of calibrating the Electrostatic Wattmeter on alternating current was explained. In connection with this work standard resistances were shown giving a two-volt drop with currents of 1000 to 2000 ampères.

A demonstration was also given of the procedure followed in carrying out life tests of electric glow-lamps in accordance with the specification drawn up by the Engineering Standards Committee. The method of setting up lamps at a standard value of watts per candle was shown, as well as the arrangements for determining and regulating the voltage of lamps under life test. The equipment includes a new and specially designed photometer on the substitution principle.

In the Metallurgy Department Mr. ROSENHAIN exhibited the process of quenching metals *in vacuo* in the ingeniously simple apparatus devised by him. The specimen is heated in a silica tube-furnace, connected at one end through a large receiver to the air-pump, and at the other through a stopcock to a tube dipping under water to a measured depth. On opening the stopcock the water rushes through as far as the receiver only, "quenching" the specimen, and washing it along to the colder parts of the silica tube.

Mr. Rosenhain also showed the ultra-violet apparatus for photomicrography which has been installed at the Laboratory. The difficulty of focussing which always obtains is greatly increased in the use of the apparatus for metallurgical work, owing to the small amount of light which appears to be obtainable by reflection from the surface of metal specimens. The method adopted, which involves the use of monochromatic blue light, with auxiliary lenses, preceding the transfer to the ultra-violet rays obtained from cadmium electrodes, was explained, and some excellent photographs were shown obtained at a magnification of 400 diameters.

In the Electricity Department Mr. CAMPBELL showed the measurement of small self-inductances down to a few tenths of a microhenry. The apparatus is arranged so that tests can be made either at low or high frequency (100 and 2000 $\sim$ per sec. respectively). In the first case a wire interrupter and a vibration galvanometer are used, and in the second a microphone hummer. A tuned telephone apparatus for the absolute measurement of the high frequencies used in wireless telegraphy was also shown. It consists of the usual arrangements for producing high frequency oscillations, and a special camera with a quickly rotating mirror, by which photographs of the oscillatory sparks can be obtained.

In the new Metrology Building Mr. JEFFCOTT showed the apparatus employed for the various measurements of length, comparisons and calibration of scales, comparison of end gauges with the standards, measurements of screws, &c. Of special interest may be mentioned the Hartmann automatic comparator, which compares two end gauges, automatically recording the readings on a revolving drum, with a magnification of 2000. The gauges are themselves automatically interchanged. The Pratt and Whitney machine for comparing end gauges was also shown, fitted with a Newell spirit-level as a contact indicator, enabling differences to be measured to 0·00001 inch. The building has been designed specially to accommodate a 50 metre comparator for the verification of surveying tapes and wires, both on the flat and in catenary, and the erection of this apparatus will shortly be completed.

In the Thermometry Department the main new feature was the newly equipped room for the testing of mercury thermometers. The old chemical laboratory has been adapted for this purpose.

A series of special comparison-tanks, designed by Dr. HARKER and Mr. W A. PRICE, has been installed on an iron railing running down the centre of the room. They are each fitted with electric heating and a system of pipes for hot and cold water supply, automatic overflow and waste, and are stirred continuously by a suitable motor.

The electric heating is so arranged for all ordinary purposes that the requisite energy can be obtained for maintaining a constant or slowly rising or falling temperature, without the use of large and expensive series resistances, while 2500 watts is available in each bath for rapid heating, between the individual sets of comparisons.

Ice and steam baths of various types, and oil and nitrate baths for high temperature work are also installed.

In the new electric-furnace room a large furnace equipment for the testing of optical pyrometers was shown in action though not yet complete. The pyrometers, carried on a massive rotating support, can be successively brought under the mouth of a furnace giving "black-body" radiation at temperatures up to 1400° C., the furnace temperature being measured by appropriate thermo-couples. A similar furnace for much higher temperatures is in preparation to be fitted in a second cell on the same elevated platform. Various types of instruments and furnaces were exhibited, and the apparatus employed by Messrs. Harker and Sexton in their determination of the effect of pressure on the sulphur boiling-point was shown in action by Mr. Sexton.

CORRESPONDENCE.

WOMEN AND THE FELLOWSHIP OF THE CHEMICAL SOCIETY.

To the Editor of the Chemical News.

Sir,—We venture to ask for the hospitality of your columns in order to make a statement of some importance, in view of the announcement made by the President of the Chemical Society of the large majority of the Fellows who are in favour of the admission of women to the Society (*Proc. Chem. Soc.*, xxiv., 203).

Four years ago a memorial was presented to the Council of the Chemical Society praying for the admission of women to the Fellowship of the Society. This memorial bore the signatures of nineteen women, all of whom were lecturers or demonstrators in chemistry in university colleges, or actively engaged in original chemical investigations. The Council at that time were unable to take any steps in the matter, but promised that the memorial should not be lost sight of in any further action that might be taken (*Proc. Chem. Soc.*, 1905, xxi., 103).

The question having been raised again by the presentation of a petition signed by 312 Fellows in June last, we communicated in July with our co-signatories of the 1904 memorial, and with other women of equal repute as chemists, in order to ascertain how many women at the present time desire the privileges afforded by Fellowship of the Chemical Society.

We have received replies from twenty-eight women, all of whom are of similar standing and possessing similar qualifications to the original signatories, expressing their interest in the present movement and their intention of at once becoming candidates for admission to the Fellowship of the Chemical Society if the Council should reach a favourable decision in this matter.

From rumours that have reached us, there appears to be some uncertainty in the minds of some Fellows of the Society as to the number of women who are prepared to avail themselves of the first opportunity of seeking the Fellowship, and we hope that the publication of the above statement will remove all misunderstanding on this point.—We are, &c.,

Ida Smedley,
M. A. Whiteley.

November 9th, 1908.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 14, October 5, 1908.

Microchemical Reactions of Arsenic Applicable in Forensic Chemistry.—G. Denigès.—A drop of the arsenical solution is slowly evaporated to dryness. The diameter of the drop must not exceed 5 mm. The residue is allowed to cool, and a drop of reagent is deposited on it; after three minutes the preparation is examined under the microscope. With ammoniacal silver nitrate red rhombic or hexagonal crystals are obtained; with acetic acid and silver nitrate crystals derived from a rhomboid dodecahedron are observed, while with magnesia mixture crystals in the shape of an X or like fern leaves are formed. If the process is performed exactly according to the directions given by the author this method may be used to obtain very characteristic crystals with traces of arsenical products present as arsenic acid.

Active Pinonic and Pinic Acids.—Ph. Barbier and V. Grignard.—The oxidation of *l*-pinene by potassium permanganate gives *l*-pinonic acid of formula $C_{10}H_{16}O_3$. When treated with hydroxylamine chlorhydrate and potash in aqueous solution it is transformed into a mixture of oximes, one dextro- and the other lævo-rotatory, which can easily be separated by taking advantage of their different solubilities in ether. By the oxidation of *l*-pinonic acid with sodium hypochlorite or hypobromite an oily acid is obtained, the formula of which is $C_9H_{14}O_4$. It is dextrorotatory, and appears to be *d*-pinic acid. *D*-pinene on oxidation gives a mixture of *d*-pinonic acid and the racemic acid, the constituents of which may be separated by crystallisation in a mixture of ether and ligroin, in which the racemic acid is only slightly soluble. By oxidising the *d*-acid the same *d*- and *l*-oximes are obtained. These researches show that the acid obtained by Tiemann by oxidising sodium *a*-campholenate, and by distilling α-dioxydihydrocampholenic acid, and described as *l*-pinonic acid, is not the true *l*-acid derived from *l*-pinene. It differs from it in melting-point, in the melting-point of its oxime, and in the products of oxidation. Possibly these two *l*-acids are the *cis* and *cis-trans*-isomers, the existence of which is foretold by theory.

Determination of Succinic Acid in Presence of Fixed Acids in Wines and Fermented Liquids.—Emm. Pozzi-Escot.—A known volume of liquid is neutralised by ammonia and acidified with a slight excess of acetic acid, and then barium chloride is added to precipitate oxalic and mineral acids. The liquid is filtered and the filtrate is boiled; excess of lead acetate is then added to precipitate albumenoids, tannins, &c., which are separated off by filtration. The filtrate is treated with sulphuric acid, then with sulphuretted hydrogen, filtered and boiled to expel excess of sulphuretted hydrogen. To separate the malic and succinic acids in the solution it is acidified with sulphuric acid and oxidised with potassium permanganate. The succinic acid remains in the solution, which is boiled; a little potassium bisulphite is added and a large excess of barium chloride to remove the sulphuric acid. The filtered liquid is concentrated, ammonia, alcoholic barium bromide, and three volumes of alcohol are added, and thus barium succinate is precipitated. This is ignited in a muffle, and the barium carbonate determined by titrating with decinormal hydrochloric acid.

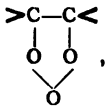
No. 15, October 12, 1908.

Method of Calculating Atomic Weights.—Louis Dubreuil.—The experimental errors which may occur in atomic weight determinations are of three kinds. (1) Errors due to the method employed; (2) errors due to the technique of the experimenter; (3) accidental errors. These last may easily be eliminated by taking the mean of a great number of experiments. The elimination of the others is much more difficult. The method of calculation used since the time of Berzelius consists in considering the atomic weights of the elements implicated in an experiment as known, except one which is calculated as a function of the others; thus the first two classes of errors are accumulated in the result. Hinrichs has recently established an important relation for the calculation of the atomic weights, which, however, is affected by a small error. Taking this

relation as a basis the author has eliminated the small error by deducing the variations of the true values of the atomic weights from the values usually adopted.

MISCELLANEOUS.

Ozonides of Simple Olefines.—C. Harries and Karl Haeffner.—Gaseous olefines explode when they come in contact with ozone, even at a very low temperature. But if the hydrocarbons of low boiling-point are diluted with a solvent which boils at a low temperature and is indifferent towards ozone, they can readily be transformed into ozonides. To convert gaseous propylene into ozonide ethyl chloride may be used as solvent, and by a modification of the method the ozonide of ethylene can, no doubt, be prepared. The ozonides are stable oils; the propylene compound crystallises at a low temperature. They may be distilled *in vacuo* without undergoing decomposition, though the operation is dangerous as slight superheating leads to an explosion. Two modifications of the ozonides appear to exist, one a thick oil and the other a mobile liquid; the latter results when the first form is distilled or heated in a vacuum. The two forms differ in refractive index and specific gravity. They do not appear to be polymers, but the thick oil seems to contain more oxygen than the other form, which is the normal ozonide. The values obtained for the molecular refraction and dispersion seem to show that the three oxygen atoms are not bound as shown by the formula—



but that one oxygen forms part of a carbonyl group, and two others are ether oxygens.—*Berichte*, xli., No. 13.

MEETINGS FOR THE WEEK.

Friday, 13th.—Physical, 5 (at the Physics Laboratory, University College, Gower Street, by kind invitation of Prof. F. T. Trouton, F.R.S.). "The Photo-electric Properties of Potassium-sodium Alloys," by J. A. Fleming "Re-combination of Ions in Air," by P. Philiips. "Influence of Magnetic Field on the Photographic Patterns produced by the Electric Spark" and Exhibition of an Experiment Illustrating an Anomalous Effect of Lagging Thin Wires, by A. W. Porter. "Rate of Growth of Viscosity in Congealing Solutions," by A. O. Rankine. "Isomers of Adsorption by Solid Surfaces from Solutions," by F. T. Trouton.

Wednesday, 18th.—Royal Society of Arts, 5. Inaugural Address of the 155th Session, by Sir William H. White, K.C.B., LL.D., F.R.S., Vice-President and Chairman of the Council.

Thursday, 19th.—Chemical, 8.30. "Meteloidine, a New Solanaceous Alkaloid," by F. L. Pyman and W. C. Reynolds. "Affinity of certain Alkaloids for Hydrochloric Acid" and "Affinity Constants of Bases as Determined by Methyl-orange," by V. H. Veley. "Relation between Absorption Spectra and Chemical Constitution—Part XII., Some Aminoaldehydes and Ketones of the Aromatic Series," by E. C. C. Baly and Miss E. G Marsden. "Organic Derivatives of Silicon—Part VIII., The Resolution of *dl*-Sulphobenzylethylisobutylsilicyl Oxide and the Properties of the Optically Active Acids," by B. D. W. Luff and F. S. Kipping. "Study of the Constitution and Properties of the Rhodanides of Inorganic Radicles," by A. E. Dixon and J. Taylor. "Action of Phosphorus Pentachloride on the Methylene Ethers of Catechol Derivatives—III., The Cyclic Carbonates of Dichloro-ethyl- and Propyl-catechol," by G. Barger. "Synthesis of Thionaphthen Derivatives from Styrenes and Thionyl Chloride," by G. Barger and A. J. Ewins.

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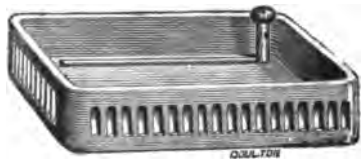

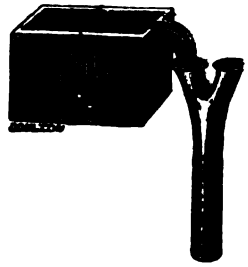

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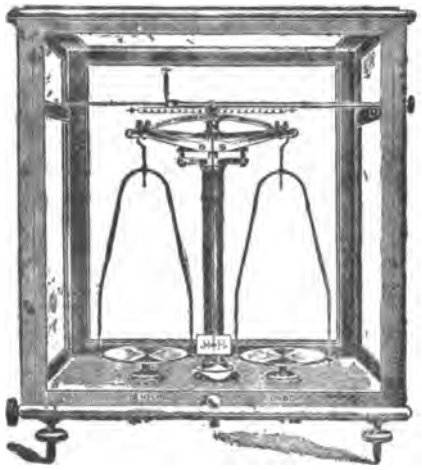

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THE CHEMICAL NEWS.

Vol. XCVIII., No. 2556.

A SIMPLE COLORIMETRIC METHOD FOR THE DETERMINATION OF FORMALIN IN MILK.

By E. W. T. JONES, F.I.C.

Reagent.—Dissolve 0·25 grm. of pure iron wire in HCl, oxidise with HNO_3, precipitate with ammonia, filter, well wash the precipitate, then dissolve it in 500 cc. of strong HCl.

Standard Formalin Solution.—Add 1 cc. of formalin (40 per cent CH_2O) to distilled water, and make to 1000 cc. 0·1 cc. of this from a 1 cc. pipette divided into tenths, added to 10 cc. of milk, equals 1 part of formalin in 100,000 parts of milk.

Procedure.—Put 10 cc. of the milk containing formalin into a large test-tube (1 inch by 6 inches), also put two more 10 cc. of pure milk into two other tubes; add to one of the latter 0·1 cc. and to the other 0·2 cc. of the dilute formalin solution; now add to each of the three tubes 10 cc. of the acid reagent, well mix, then put all the tubes simultaneously into water at 90° C. and keep at 80° to 85° C. for twenty to twenty-five minutes; then add 30 cc. of cold water to each tube, well shake, cool and throw on to filters; when through pour on to each of the filters about 10 cc. of cold distilled water, and when this is through make each of the blue filtrates to 100 cc. If the sample being tested, compared in a Nessler glass, gives a darker colour than the 0·1 cc. standard, it is better to dilute the 0·2 cc. standard to 200 cc., when 100 cc. will equal 0·1 cc., which is dark enough to use as a standard for comparison.

I find with only 0·1 cc. of the dilute formalin in 10 cc. of milk the colour is not so pure a blue as the diluted solution of a larger amount, and therefore for nesslerising the larger amounts it is better to dilute the stronger for a standard.

I do not describe the nesslerising because every laboratory assistant knows this procedure.

N.B.—The sample and the standards must be submitted to strictly the same conditions side by side.

I have obtained regularly most satisfactory results. The great advantage is that the determination can be made with only 10 cc. of milk in an hour or less, and I believe with greater accuracy than by the more troublesome distillation process requiring 100 cc. of milk.

Wolverhampton, October 30, 1908.

THE PRESENT STATE OF OUR KNOWLEDGE OF RADIO-ACTIVITY.*

By A. DEBIERNE.

(Continued from p. 236).

III.

We have seen that radio-active substances are characterised by the spontaneous emission of a special radiation; I shall now describe the principal properties of this radiation.

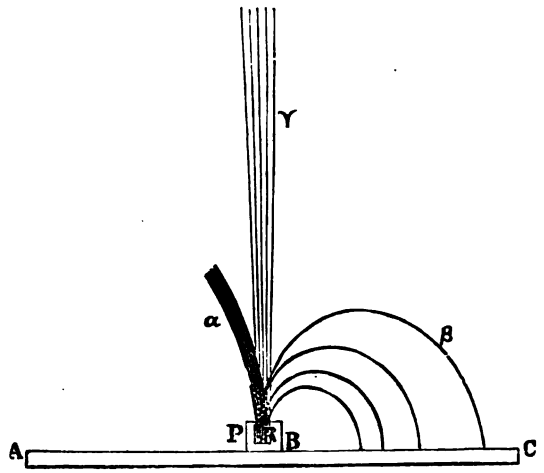
The complexity of the radiation emitted by radium salts was shown by the experiments of Becquerel, P. Curie and Mdme. Curie, Rutherford, Villard, &c. It is possible to distinguish different kinds of rays, which may be compared to those obtained by an electric discharge in a Crookes tube.

The existence of three kinds of rays has been been proved in this tube—cathode rays, which are normally projected from the negative electrode; canal rays, which are found behind the cathode, when the latter is perforated; and Röntgen rays or X-rays, which are emitted chiefly by a sheet of metal, called the anticathode, placed in front of the cathode. It has been shown that the cathode rays are composed of particles much smaller than the atoms, and negatively charged; they are called electrons. The velocity of these particles is of the order 100,000 km. per second. The canal rays are formed of much larger particles, postively charged, and having a rather smaller velocity. Finally, the Röntgen rays are not electrified; they appear to be a peculiar disturbance in the ether produced every time an electron with a high velocity is suddenly stopped by a solid substance.

The rays emitted by active substances may also be grouped in three classes:—The α-rays, positively electrified, and analogous to the canal rays; β-rays, negatively electrified and analogous to the cathode rays, and the γ-rays, non-electrified and analogous to the Röntgen rays.

The electric charges emitted by the radium rays were first directly determined by Pierre Curie and Mdme. Curie.

The distinction between the different kinds of rays could be established by different methods. Firstly, their different powers of penetrating matter could be employed. The α-rays have the least penetrating power; they are completely absorbed by a layer of air a few centimetres thick



or by a sheet of aluminium one-thirtieth mm. thick. β-Rays of different penetrations are known, but, in general, they are much less penetrating than α-rays; some of them can pass through aluminium 1 cm. thick. Finally, the γ-rays have very great power of penetration, and it has been shown that they are not completely absorbed by a screen of lead several cm. thick.

The different kinds of rays may also be distinguished by the action of a magnetic field. For the projection of electrified particles may be compared with an electric current, and an intense magnetic field may exert on the moving particles a force great enough to modify their trajectory. The β-rays are the most sensitive to the action of the magnetic field; the α-rays undergo a much smaller deviation for the same field, and in the opposite direction. The γ-rays are not affected. The action of a magnetic field upon a pencil consisting of the three kinds of rays may be illustrated as in Fig. 1. The magnetic field produced by a field magnet, for example, being normal to the plane of the paper and directed towards this plane, the α-rays are slightly deflected to the left, and the β-rays are strongly deflected towards the right.

The trajectory of the electrified particles of the α- and β-rays can also be modified by an electric field, as, for example, by making them pass between the plates of an

* *Revue Générale des Sciences*, 1908, xix., 691.

air condenser, but the deviation obtained is always very slight. The α-rays are, of course, attracted towards the negatively charged plate and the β-rays towards the positively charged plate.

It may easily be shown that the electric and magnetic deviations must depend on the electric charge on each particle, on its mass, and its velocity; if for a certain kind of rays the deviations in a field of known intensity are measured, the velocity of the electrified particle can be calculated, and also the ratio e/m of the charge on a particle to its mass m.

These measurements have been made for the different kinds of rays. The measurements for the β-rays have been made chiefly by H. Becquerel and Kauffmann. They have shown that the β-radiation emitted by radium includes rays of very different velocities, from a velocity about half that of light to a velocity nearly equal to that of light; *i.e.*, 300,000 km. per second.

According to Kauffmann's experiments the most rapid rays give a value of e/m considerably smaller than that corresponding to the less rapid rays. The latter value appears to be identical with the value of e/m for the cathode rays, the velocity of which is still smaller. In modern theories of the nature of electricity, an interpretation of the greatest importance has been placed upon the variation of the ratio e/m with the velocity of the β-particle, for it has been shown that the material aspects of the electron—motion, inertia, kinetic energy—can be explained perfectly by considering only the electric charge in rapid motion in the ether, and that the apparent mass which results from this motion would increase rapidly when the velocity approached that of light. According to all the experiments which have been performed the β-ray must be regarded as a cathode ray of great velocity produced by an electron in rapid motion.

Measurements of the magnetic and electric deviations of the α-rays have been made chiefly by Rutherford. They show, to a first approximation, that all the α-particles have the same value for e/m in the rays emitted by different active substances — radium, thorium, actinium, and polonium—and if it is allowed that the electric charge e is equal, and of opposite sign to that of the electron, the mass of each particle is twice that of an atom of hydrogen.

As I have mentioned, the γ-rays do not suffer any deflection by a magnetic or electric field.

The different kinds of rays may cause the phosphorescence of certain substances—salts of the alkalis and alkaline earths, glass, barium platinocyanide, zinc sulphide, certain minerals, willemite, kunzite, diamond, &c., and also certain organic substances, paper and cotton. I have recently shown that the skin of the hands also becomes luminous under the action of radium rays. However, all these substances are not equally sensitive to the different rays; thus, barium platinocyanide is very sensitive to the γ-rays and zinc sulphide to the α-rays. The phosphorescence of zinc sulphide under the action of the α-rays exhibits a very curious phenomenon. When the phosphorescent substance, which has been subjected to the rays, is examined by the aid of a magnifying glass, it is seen that it is not uniformly luminous, but that there are on its surface numbers of little luminous points which appear and disappear very rapidly; thus an impression is produced of a multitude of little sparks on the zinc sulphide. It is thought that each little luminous point is produced by the bombardment of an α-particle, and attempts have been made to determine the number of particles emitted per second by an active substance by observing the number of luminous points which appear in the same time on a zinc sulphide screen. However this may be, this phenomenon of scintillation appears to be a characteristic property of the α-rays.

The path of the different kinds of rays can be followed by observing the phosphorescence, and thus the deviation in a magnetic field, and also the passage of the rays through different screens can be determined.

The path of the rays can be determined more exactly by using the photographic plate. This method, which was specially employed by Becquerel, enables us to verify the shape of the path of the β-rays in a magnetic field and various other properties.

The α-, β-, and γ-rays ionise gases, *i.e.*, they make them conductors by generating throughout them positive and negative charges, which move in an electric field; by the ionisation produced in a gas the intensity of the radiation of an active substance can be measured. The α-rays generally produce the greatest ionisation; as these rays are very easily absorbed, it follows that ionisation by an acive substance is greatly diminished by a screen, even at very thin one. The absorption of the rays by matter has been the subject of much important work which has given very interesting results, especially for the α-rays.

In the first place, it has been proved by Mdme. Curie that in air the α-rays have a range which comes to an abrupt end, and that at a certain distance from the active substance α-rays can no longer be detected. For instance, with polonium they disappear at 4 cm. and with radium at 7 cm. Bragg and Kleemann have studied this phenomenon, and have suggested a very satisfactory theory to account for it.

They have shown that every active substance (and we shall see further on that a great number of active substances must be supposed to exist) emits α-particles of the same kind and having the same velocity. If the active substance were infinitely thin, all the particles would have exactly the same range in air; this range is a quantity which is characteristic of the kind of α-rays and also of the nature of the active substance.

Rutherford has shown that the phenomenon of scintillation and photographic action cease at the same distance as the ionisation of a gas, and that at this distance the velocity of the α-particle is more than 40 per cent of its original value.

Finally, it must be mentioned that besides the rays previously described certain active substances emit electrons with very low velocities, and that the β- and γ-rays, when they impinge on matter, cause the emission by it of secondary rays analogous to those which the Röntgen rays produce.

IV.

Before discussing new phenomena the study of which has led to present-day theories of radio-activity, I must give an account of the general hypothesis, made at the beginning of their researches by Pierre Curie and Mdme. Curie, to explain the peculiar properties of radio-active substances.

The principal point to explain is the spontaneous and continuous emission of energy; and as we shall see presently, the quantity of energy emitted is much larger than might at first be supposed.

The two following hypotheses have been proposed:—

In the first hypothesis it is supposed that the energy is supplied from external sources in the form of a special radiation. This radiation, the nature of which is unknown, would cause in radio-active substances a phenomenon analogous to that which is produced with phosphorescent substances; it would be absorbed by the active substance, and its energy given back to the surroundings in the form of α-, β-, and γ-rays, &c.

In the second hypothesis it is supposed that the energy is derived from the active substance itself; the emission of energy must then be accompanied by a modification of the substance. Pierre Curie and Mdme. Curie, who had demonstrated the atomic nature of the new property, made the bold supposition, even before the discovery of radium, that in this case the transformation would take place in the radio-active atom itself, which would thus undergo a veritable transmutation yielding different atoms. One of the most important consequences was also pointed out, *i.e.*, the progressive disappearance of the radio-active element, which would give place to other elements.

This hypothesis of atomic disintegration also explains the emission of material particles having high velocities.

It is not impossible to combine the two hypotheses and to suppose that an atomic transformation occurs, produced by an external agency of unknown nature acting continuously on the active substance.

As is well known, the hypothesis of atomic transmutation is the most fruitful, and has served as the basis for modern theories.

(To be continued).

JUBILEE OF PROFESSOR SAKURAI.

Professor Sakurai completed the twenty-fifth year of his professoriate in the Tokyo University last year, and this event has recently been made an occasion of public celebration by his friends and former pupils, who among other things have raised a considerable sum of money to be used as a fund for the encouragement of chemical research in association with his name and under the direction of the Tokyo Chemical Society. A volume of papers embodying scientific work recently done by some of his former pupils, together with those by Professors Kuhara and Haga, have also been specially contributed on this occasion to celebrate Professor Sakurai's jubilee. From this volume we have much pleasure in reprinting two papers by Masataka Ogawa on some new elements. The first of these is given below.

PRELIMINARY NOTE ON A NEW ELEMENT IN THORIANITE.*

By MASATAKA OGAWA, Rigakushi.

While working on thorianite in University College, London, under Sir William Ramsay's direction a few years ago, an element believed to be new was met with in the iron group in the usual course of analysis, but, owing to the small quantity, its nature could not be fully established. The subject has been resumed since my return to Japan, and it has fortunately been found that the same element also occurs in other minerals, such as reinite and molybdenite, both found in this country. This somewhat incomplete paper is now published as a preliminary notice of the work, which is still going on.

Treatment of Thorianite.

Preparation of the Oxide.—Finely powdered thorianite was treated with boiling concentrated nitric acid and the solution decanted off, this process being repeated until the fresh nitric acid was no longer coloured yellow. The residue, amounting to about 3 per cent and now nearly free from the chief constituents of thorianite, viz., thorium, uranium, iron, and lead, was boiled with concentrated hydrochloric acid, by which treatment a considerable quantity of titanium and antimony passed into solution and was decanted off. The residue was then treated with hydrofluoric acid in the presence of concentrated sulphuric acid in order to remove the silica, and, after ignition and fusion with hydrogen potassium sulphate, the whole mass was dissolved in cold water.

On boiling the diluted solution, however, a slight turbidity occurred, which was found to be due to the separation of a basic titanium compound. Hydrogen sulphide was then passed through the filtered solution, and some brown precipitate thereby formed was filtered off. To the filtrate ammonia was added after oxidation with nitric acid, and the precipitate formed was dissolved in dilute hydrochloric acid. To this solution oxalic acid was added, which produced a small quantity of a white precipitate consisting of the oxalates of thorium, &c., and this was filtered off. Ammonia and ammonium sulphide were then added to the filtrate; the black precipitate thereby formed was dissolved in dilute hydrochloric acid and re-precipitated by ammonium sulphide in the presence of an excess of ammonium carbonate, thus keeping in solution any uranium which might still be present. The ammonium sulphide precipitate was again dissolved in dilute hydrochloric acid and re-precipitated by ammonia in the presence of an excess of ammonium chloride. The precipitate, consisting chiefly of the hydroxides of iron, aluminium, and zirconium, was dissolved in dilute hydrochloric acid, the solution nearly neutralised with sodium carbonate, and boiled after the addition of sodium thiosulphate. The precipitate now formed was dissolved in hot concentrated hydrochloric acid, the separated sulphur filtered off, and the solution re-precipitated by sodium thiosulphate, the usual precautions being taken.

The precipitate was once more dissolved in concentrated hydrochloric acid, and, after evaporating off the excess of the acid and heating with some sulphuric acid, a saturated solution of potassium sulphate was added to the solution. After standing for twenty-four hours, the double sulphate of zirconium and potassium, which was formed, was filtered off. The filtrate was evaporated to a small bulk, and the crystals formed of potassium sulphate and alum were removed. After mixing the greenish coloured mother-liquor with 90 per cent alcohol and filtering, ammonia was added to the solution, when a pale yellow precipitate, believed to consist of the hydroxide of the new element, was obtained.

Properties of the Oxide.—The hydroxide above obtained is insoluble in ammonium chloride. It dissolves in alkalis, but not so easily as aluminium hydroxide. On drying at 100°, it becomes nearly black, and on ignition it changes to a dark brown powder. The ignited oxide is insoluble in acids, even in hot concentrated sulphuric acid to which some hydrofluoric acid has been added. On fusion with hydrogen potassium sulphate, however, and extracting the fused mass with water, the oxide may again be brought to solution. On fusion with sodium carbonate and potassium nitrate, the oxide gives a green mass, having much the same appearance as that of an alkaline manganate. This dissolves in water and forms a green coloured solution, from which a current of carbonic acid produces a brown precipitate, soluble in hydrochloric acid.

A solution of the hydroxide in hydrochloric acid has a yellowish green colour and gives a chocolate-brown precipitate on boiling with sodium thiosulphate. The same solution gives a greenish black precipitate with ammonium sulphide, insoluble in an excess of the reagent, and a yellowish brown precipitate with potassium chromate, insoluble in acetic acid and quite different in appearance from that of lead chromate. Potassium iodide gives no precipitate.

The yield of the oxide was a few centigrms. from one kilgrm. of the mineral. The portion of the mineral soluble in nitric acid also yielded a small quantity of the oxide.

I had also in store all the ammonia precipitate separated from the crude hydrogen sulphide precipitate given by Sir William Ramsay. The whole of this precipitate was ignited and treated as if it had been the original mineral, and a little over one decigrm. of the oxide was obtained from this source.

Salts.—Several attempts have been made to get well crystallised salts, but thus far without success. After fusing the oxide with hydrogen potassium sulphate, dissolving the fused mass in water, precipitating the hydroxide by ammonia, dissolving the precipitate in different acids, and evaporating these solutions, very soluble imperfect crystals were, indeed, obtained; but these crystals change to basic insoluble compounds on treatment with water. Attempts to get well defined crystals of double salts, containing the element in question, also ended in failure.

Anhydrous Chloride.—The oxide was heated in a porcelain boat in a current of chlorine gas, saturated with the vapour of carbon tetrachloride. There was formed a

* *Journal of the College of Science, Imperial University, Tokyo, Japan*, vol. xxv., Article 15.

very slight white sublimate, the main portion of the product remaining in the boat as a white powder having a slightly yellow tinge. On continued heating at low redness, a pale green sublimate was formed; but it was very slow in formation and insignificant in amount.

The residue in the boat, doubtless consisting of the anhydrous chloride, became deep green on being moistened with water, and dissolved into a pale green solution on the addition of more water, acidified with nitric acid. A portion of the solution was tested for thorium with negative results.

Equivalent Weight.—(1) A known weight of the chloride, prepared as above and contained in a small tube, was dissolved in acidified water, precipitated by ammonia, and the precipitate was ignited and weighed as oxide. 0·1092 grm. of the chloride gave 0·0742 grm. of the oxide, from which the number 50·3 was calculated as the equivalent weight of the element. (2) The oxide was fused with hydrogen potassium sulphate, dissolved in water, and precipitated by ammonia. This precipitate was dissolved in dilute sulphuric acid, the solution evaporated to dryness, and heated over a sulphur-bath until the weight became constant. The residue was ignited and weighed as oxide. 0·1253 grm. of the sulphate gave 0·0747 grm. of the oxide, again leading to the value 50·3. (3) The chloride, of which 0·0652 grm. was taken, was dissolved in water, acidified with nitric acid, and precipitated by silver nitrate, which gave 0·1120 grm. of silver chloride, whence follows the number 48 as the equivalent weight of the element.

Judging from the mode of working, the properties of the oxide and the chloride, as well as from the equivalent weight, the element in question must be considered as hitherto unknown, and this view has been further confirmed by—

Spectroscopic Examinations.—The oxide was fused with sodium carbonate and potassium nitrate, the fused mass dissolved in water, and the brown precipitate, obtained by passing a current of carbonic acid through the solution, was dissolved in dilute hydrochloric acid, and subjected to spectroscopic examinations. The lines were not remarkably strong, but three lines could be observed in the green-blue part of the spectrum. There were no manganese lines. Of the three lines, the strongest was the one having the wave-length 4882, believed to be correct within 10 units. The other two lines could also be measured, but, owing to their feebleness, the statement of their wave-length is here reserved.

Examination of Reinite.

The finely powdered mineral was fused with a mixture of sodium carbonate and potassium nitrate, the fused mass treated with water, ferric oxide filtered off, and the filtrate was saturated with carbonic acid gas. A brown precipitate, containing a little iron and silica, but otherwise apparently the same as that obtained, under like conditions, from the oxide extracted from thorianite, was produced. This was dissolved in dilute hydrochloric acid, and examined spectroscopically. The spectrum was exactly the same as before, though other lines were observed in addition.

Another portion of the powdered mineral was digested with hot nitro-hydrochloric acid, until its brown colour was completely changed into yellow, the solution evaporated to dryness, and the residue extracted with water acidified with hydrochloric acid. The solution, separated from insoluble tungstic acid, was saturated with hydrogen sulphide, the black precipitate thereby formed was filtered off, and the filtrate, after evaporation and oxidation, was treated with ammonia. The hydroxides of the iron group thus precipitated were re-dissolved in dilute hydrochloric acid, boiled with sodium thiosulphate in order to remove iron, and the re-precipitated hydroxides were filtered and ignited. The ignited oxides were then heated in a current of chlorine gas and carbon tetrachloride vapour. A difficultly volatile chloride, apparently identical with that obtained from thorianite, was thus produced. Its spectrum, also, was exactly the same as before. The yield of the oxide was, however, very much better, about 1 decigrm. having been obtained from 200 grms. of the mineral.

Examination of Molybdenite.

The powdered mineral was roasted in air until sulphurous acid gas was no longer given off. The residue, which was yellow when hot and pale greenish yellow when cold, was digested with a dilute solution of ammonia. The portion insoluble in ammonia was then digested with dilute hydrochloric acid, and the solution filtered from some undecomposed portion of the mineral. The acid solution was nearly neutralised with ammonia, and, after saturation with ammonium chloride and on standing, a pale yellow precipitate was formed. The precipitate, apparently ammonium vanadate, was removed by filtration. To the yellowish green filtrate, hydrogen sulphide was passed to saturation, and the black precipitate thereby formed was filtered off. The filtrate was freed from hydrogen sulphide by boiling, and then ammonia and hydrogen peroxide were added. The precipitated hydroxides were re-dissolved in dilute hydrochloric acid and boiled, as before, with sodium thiosulphate in order to remove iron.

The re-precipitated hydroxides were filtered, ignited, and fused with a mixture of sodium carbonate and potassium nitrate, and the fused mass dissolved in water. After decomposing the solution with hydrochloric acid, ammonia gave only a slight turbidity, due apparently to the presence of a small quantity of aluminium. On reduction, however, with zinc and hydrochloric acid, the solution gave an abundant precipitate with ammonia in the presence of an excess of ammonium chloride. The spectrum of the hydroxide thus obtained and dissolved in hydrochloric acid showed the same lines as those observed in the case of thorianite and reinite. The zinc used for reduction was prepared by the electrolysis of a pure solution of zinc ammonium sulphate.

The ammoniacal solution obtained on treating the roasted mineral with ammonia was also found to contain a portion of the element in question originally existing in the mineral; for this solution, after acidification with acetic acid and the removal of molybdic acid as barium molybdate, gave no precipitate with ammonia, but, on reduction with zinc and hydrochloric acid, ammonia in the presence of ammonium chloride produced some precipitate from it, which, on examination, was found to be the same hydroxide as that obtained, under like conditions, from the hydrochloric acid solution of the roasted mineral.

Thorianite, Reinite, and Molybdenite.

It will be seen from what has been stated that the new element is contained in reinite and molybdenite, as well as in thorianite, and, although it cannot yet be definitely stated, it is almost certain that molybdenite contains it in even a larger proportion than reinite.

It may be remembered that Dr. E. H. Buchner, in the course of his analysis of thorianite, observed the precipitation of a small quantity of a black sulphide in the arsenic group and occurrence of two other unknown substances in the bismuth-cadmium group (CHEMICAL NEWS, 1906, xciv., 233). I have not only confirmed his observations with thorianite, but have also met with the same unknown substances in the course of my examination of reinite and molybdenite. It is a striking case of coincidence that the three different minerals should contain these same unknown substances, besides the new element which forms the subject of this paper.

Conclusions.

Thiorianite, reinite, and molybdenite contain, in common, a new element, which is characterised by the following properties:—

1. The hydroxide, precipitated by ammonia in the presence of an excess of ammonium chloride, has an almost white colour with a pale yellow tinge. It is soluble in alkalis, but not so readily as aluminium hydroxide.

2. On drying at 100°, it becomes nearly black. The

ignited oxide has a dark brown colour and is insoluble in acids, but becomes soluble in water, after fusion with hydrogen potassium sulphate.

3. A solution of the hydroxide in hydrochloric acid has a yellowish green colour, and gives a chocolate-brown precipitate on boiling with sodium thiosulphate.

4. When ammonium sulphide is added to a solution of the chloride a greenish black precipitate, insoluble in an excess of the reagent is formed.

5. A solution of the chloride, acidified with acetic acid, gives a yellowish brown precipitate with potassium chromate. Potassium iodide gives no precipitate.

6. On fusing the oxide with sodium carbonate and potassium nitrate, a green mass is obtained, soluble in water. This solution gives a brown precipitate on passing a current of carbonic acid gas, the precipitate readily dissolving in dilute hydrochloric acid.

7. After fusion in the above manner, neither ammonia nor ammonium sulphide gives any precipitate; but on acidifying the solution, to which ammonium sulphide has been added, with hydrochloric acid, a brown precipitate is formed.

8. After the reduction of the above solution with zinc and hydrochloric acid, ammonia in the presence of an excess of ammonium chloride again precipitates the hydroxide.

9. When the oxide is heated in a current of chlorine gas saturated with the vapour of carbon tetrachloride, a difficultly volatile anhydrous chloride is obtained, which dissolves in water to form a pale green solution.

10. The chloride gives a characteristic line, besides two other feebler lines, in the green-blue part of the spectrum, having a wave-length of 4882.

11. The equivalent weight of the element is about 50; this element probably filling the gap between molybdenum and ruthenium and having an atomic weight of about 100.

12. The element appears to exist in two degrees of oxidation. The lower oxide behaves as a basic oxide, and comes along with alumina in the ordinary course of analysis. Its separation from the latter is, indeed, very difficult, the conversion into the difficultly volatile chloride by heating in a current of chlorine gas and carbon tetrachloride vapour being, at present, the only means of separation. The higher oxide behaves as an acidic oxide, similar to molybdenum trioxide, and is reducible into the basic oxide by means of zinc and hydrochloric acid.

The name Nipponium, with the symbol Np, was once suggested by Sir William Ramsay, and this name will be provisionally employed to indicate the new element.

I cannot conclude without expressing my heartiest thanks to Sir William Ramsay for his kind assistance and advice, and for his generosity in supplying me with the valuable material for this research while I was working in his laboratory.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 241).

Confirmatory Experiments and References (continued).

Colour of Vanadium Solutions. — 100 mgrms. V as Na_3VO_4 were added to a little concentrated HCl, and the mixture was evaporated to a small volume: the solution was at first orange-yellow but turned greenish blue on evaporation with HCl, showing reduction of the vanadium to the quadrivalent state. Excess of concentrated HNO_3 was added and the mixture again evaporated to a small volume: the solution rapidly became yellow and the concentrated solution was deep red, doubtless owing to the presence of free vanadic acid (see below). On diluting to about 20 cc. it became yellow. The solution was neutralised with 10 per cent NaOH solution: a small yellow precipitate separated during the neutralisation, but dissolved on the addition of a little more NaOH; the alkaline solution was of a deeper yellow colour than the dilute HNO_3 solution. Na_2O_2 was added: no change was observed. The mixture was boiled for one minute; the solution became colourless. The solution was cooled and made acid with HNO_3: it became yellow, the colour being deepest at the neutral point. To a portion of this acid solution were added 50 mgrms. Ti as $TiCl_4$: no change in colour was observed, showing that the Na_2O_2 had been completely decomposed and that no pervanadate had remained in the alkaline solution after boiling. Düllberg (*Zeit. Phys. Chem.*, 1903, xlv., 172) considers that the yellow colour is due to $H_4V_6O_{17}\equiv$, an ion of the tetrabasic hexavanadic acid, $H_4V_6O_{17}$ (see C. E., *P58*a*, N. 6).

P. 52, N. 11: *Division of Zinc in the Sodium Peroxide Treatment.*—500 mgrms. Fe as $FeSO_4$ and 10 mgrms. Zn as $ZnCl_2$ were treated by P. 52, the final volume being about 30 cc.; the filtrate was tested for zinc by P. 53 and 57: only about 3 mgrms. Zn were found, showing that the remainder had been carried down with the $Fe(OH)_3$. The experiment was repeated with 5 mgrms. Zn as $ZnCl_2$: only a trace of zinc was found in the filtrate. The last experiment (with 5 mgrms. Zn) was repeated, except that a large excess of NaOH was added before the Na_2O_2: 1 to 2 mgrms. Zn were found in the filtrate. The precipitate of $Fe(OH)_3$ was analysed by P. 64, 66, 67: the remainder of the zinc was found.

500 mgrms. Mn as $MnCl_2$ and 10 mgrms. Zn as $ZnCl_2$ were treated by P. 52, and the filtrate was tested for zinc by P. 53 and 57: no zinc was found. The experiment was repeated with 20 mgrms. Zn: a very small precipitate of ZnS was obtained in P. 57 which contained less than 0·5 mgrm. Zn. On analysing the precipitate by P. 61, 64, 66, and 67 the zinc was found. Therefore nearly 20 mgrms. zinc may be completely carried down when 500 mgrms. Mn are present. The experiment was repeated with 10 mgrms. Zn, except that the acid solution was poured into a fairly concentrated NaOH solution in P. 52: about 0·3 mgrm. Zn was found in the filtrate, showing that a little zinc remains in the filtrate in this case.

For the fact that zinc is carried down with nickel and cobalt, see T. A., No. 140 to 143.

Separation of Vanadium from Manganese.—A mixture containing 250 mgrms. Mn as nitrate and 1 mgrm. V as Na_3VO_4 was treated by P. 52; the filtrate was tested for vanadium by *P. 58*e*: a very good test was obtained.

P. 53, N. 1: *Solubility of Zinc Phosphate, Carbonate, and Oxalate in Ammonium Hydroxide.*—100 mgrms. Zn as $ZnSO_4$ along with an equivalent amount of sodium phosphate, sodium carbonate, or oxalic acid were dissolved in separate experiments in about 30 cc. cold water and about 2 cc. HNO_3 (1·20). The solutions were neutralised with NH_4OH (0·96) and an excess of about 3 cc. added: clear solutions were obtained in each case.

P. 54, N. 1 to 3: *Confirmatory Test for Aluminium.*—See Knoevenagel (*Prakticum des Anorg. Chemikers*, p. 160).

0·5 mgrm. Al and 0·2 mgrm. Co as nitrates were treated by P. 54: the ash retained the form in which the filter-paper was rolled and had a brilliant blue colour. The experiment was repeated with 0·2 mgrm. Al and 0·1 mgrm. Co: the residue was distinctly blue. The experiment was repeated with 0·1 mgrm. Al and 0·1 mgrm. Co: no blue colour was apparent.

Effect of other Elements upon the Confirmatory Test for Aluminium.—0·5 mgrm. Al and 0·2 mgrm. Co as nitrates were treated by P. 54, except that 1 mgrm. Fe as $Fe(NO_3)_3$ was also added: the residue was brown. This experiment

* From the *Journal of the American Chemical Society*, xxx., No. 4.

was repeated with 1 mgrm. Al: the residue was partly blue. The experiment was repeated with 2 mgrms. Fe and 1 mgrm. Al: the blue colour could scarcely be distinguished.

The experiment with 0·5 Al was repeated in the presence of 1 mgrm. Be as $Be(NO_3)_2$: the residue was blue. The experiment was repeated with 5 mgrms. Be: a satisfactory test for aluminium was obtained. The experiment with 1 mgrm. Al was repeated in the presence of 5 mgrms. Be and also of 10 mgrms. Be as nitrate: a fair test for aluminium was obtained in the presence of 5 mgrms. Be, but none with 10 mgrms. Be. In the latter case the paper and ash disintegrated.

1 mgrm. Al as nitrate was treated by P. 54, in the presence of 1, 2, and 5 mgrms. U as $UO_2(NO_3)_2$ respectively: a slight test for aluminium was obtained in the first experiment, a very poor one in the second, and none in the third. The experiment was repeated with 0·5 Al and 1 mgrm. U: the test was very poor.

The experiment was repeated with 1 mgrm. Al and 5 mgrms. V as Na_3VO_4: the residue was distinctly blue.

0·5 mgrm. Al as $Al_2(PO_4)_3$ and 0·2 mgrm. Co as nitrate were treated by P. 54: the residue was blue.

1 mgrm. Al as nitrate was precipitated by adding to it $NaHCO_3$ solution and boiling. The precipitate was filtered off, washed once, and treated by P. 54: the ash fused together into a small mass and no blue colour was apparent. 1 mgrm. Al and about 5 mgrms. Na as nitrates were then treated by P. 54: the result was the same, showing that the failure in the first experiment had been due to the presence of a sodium salt.

50 mgrms. SiO_2 as Na_2SiO_3 were dissolved in about 2 cc. HNO_3 (1·20) and 30 cc. water, heated to boiling, and made alkaline with NH_4OH: the solution slowly became turbid, and a gelatinous precipitate separated. This was collected on a filter, washed, and a portion of hot HNO_3 (1·20) was poured through the filter two or three times: the silica did not dissolve. The HNO_3 solution was tested for aluminium by P. 54, 0·5 mgrm. Co as nitrate being added: the residue was black. The experiment was repeated except that 2 mgrms. Al as nitrate were also present: a fair test for aluminium was obtained.

P. 56, N. 1: *Test for Chromate with Hydrogen Peroxide.* or the constitution and properties of the perchromic acid, and of other perchromates, see Riesenfeld (*Ber.*, 1905, xxxviii., 1885, 3380, 4578, and 4068).

In a series of test-tubes, each of which contained 0·3 mgrm. Cr as K_2CrO_4, were placed 0, 0·5, 2, 5, 10, and 20 cc. HNO_3 (1·20); each solution was diluted to 20 cc.; 3 to 5 cc. ether and 2 cc. H_2O_2 (3 per cent) were added, and the solutions were shaken: in the solution containing no acid, and in the solutions containing 5, 10, and 20 cc. acid, no blue colour was obtained, but the remaining two solutions became blue at once, and the blue compound was extracted by the ether on shaking. In the solution containing 2 cc. HNO_3, the colour in the ether layer in contact with the solution disappeared in about half an hour on standing; the colour was more brilliant and remained for a longer time in the experiment with 0·5 cc. HNO_3. The experiment was repeated with H_2SO_4 (1·20) instead of HNO_3: no colour was obtained with no acid or with 10 and 20 cc. of it; the most brilliant and most permanent colour was obtained in the experiment with 0·5 cc. acid. The experiment was repeated with 30 per cent acetic acid: in this case the best tests were obtained in the solutions containing 10 and 20 cc. of acid; with the more delicate solutions only a faint blue colour was obtained.

Another series of experiments was performed with solutions that contained 0·3 mgrm. Cr as K_2CrO_4, and 2 cc. HNO_3 (1·20) in about 20 cc.; 3 cc. ether and varying amounts of H_2O_2 (3 per cent) were added: with 10 cc. H_2O_2 no test was obtained, with 5 cc. a slight one, with 2 cc. a good test, but with 0·5 cc. H_2O_2 a much better one.

In all of these experiments in which the blue perchromic acid was formed, the blue colour disappeared completely in less than one hour. In some of them the solution was warmed to 50 or 60°: the blue colour disappeared very rapidly. These experiments prove that perchromic acid is a very unstable substance even in the absence of an excess of H^+ or of H_2O_2 and is still more so at higher temperatures.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, October 22nd, 1908.

Sir William Ramsay, K.C.B., F.R.S., President, in the Chair.

Messrs. H. R. Lyell and C. T. Rutter were formally admitted Fellows of the Society.

The President referred to the loss sustained by the Society through the death, on August 25th, of Prof. A. H. Becquerel, who was elected an Honorary and Foreign Member in May, 1904; also of Sir Thomas Stevenson and Mr. Bennett H. Brough, who died on July 27th and October 3rd respectively.

On the question of admitting women to the Fellowship of the Chemical Society, the President announced that 1758 voting papers were received before the close of the ballot; of these there were, in favour of admission, 1094; against admission, 642; neutral, 22. The matter is now receiving the earnest attention of the Council.

The President further stated that he was authorised by the Council to inform the meeting that in all proceedings relating to the admission of women as Fellows, the Honorary Secretaries have throughout acted under the direction of the Council.

Certificates were read for the first time in favour of Messrs. Noel Charles Akers, 26, King's Avenue, Muswell Hill, N.; Maurice Barrett, Holly Bush, Headingley, Leeds; Robert Bickerstaffe, Chilterns, Wooburn Green, Bucks; William Sudderick Brakes, 137, Steade Road, Sheffield; Leonard Clement, B.A., Beamsley, Fairhaven, near Lytham; Robert Charles Cowley, College of Pharmacy and Chemistry, Brisbane; Hugh Cecil Duckworth, 66, Victoria Street, S.W.; Horace Barratt Dunnicliff, B.A., B.Sc., 39, Herne Hill, S.E.; Reginald Freeman Easton, 38, Edith Road, W. Kensington; Henry Fairburn, Northallerton; Arthur Frank Girvan, B.Sc., 8, Chatsworth Road, West Norwood, S.E.; John Henry Albert Hebron, B.Sc., 9, Buller Street, Derby; Grove Johnson, 124, Savoy Court, Strand, W.C.; Patrick Anderson Keiller, B.Sc., Colombo, Ceylon; Frank William Linch, 9, Gladstone Avenue, Manor Park, London, E.; Arthur Stanley Lindley, Prince Building, Medows Street, Bombay; Edward Pigott Minett, M.D., M.R.C.S., L.R.C.P., Guy's Hospital, S.E.; Cyril Lawrence Norman, 36, Halesworth Road, Lewisham, S.E.; Alexander Robert Normand, M.A., B.Sc., Wilson College, Bombay; Alexander Rennie, Johannesburg, Transvaal; Ezra Lobb Rhead, Stonycroft, Polygon Avenue, Levenshulme; Maurice Salamon, 64, Oxford Gardens, N. Kensington, W.; William Saunderson, B.Sc., The Armidale School, Armidale, N.S.W.; Ernest Sommerfeldt, Ph.D., The University, Tübingen; Francis George Henry Tate, Kilmersdon, Homecroft Road, Sydenham, S.E.; Robert Dickie Watt, M.A., B.Sc., P.O. Box 434, Pretoria, Transvaal; Arthur Weighell, Prince Building, Medows Street, Fort, Bombay; Frederick Williams, B.A., Bute Villa, Aberdare, South Wales; William George Winterson, B.Sc., 89, Salisbury Road, Barnet; Allister MacLean Wright, P.O. Box 617, Christchurch, New Zealand.

Of the following papers, those marked * were read :—

*199. "*The Relationship of Colour and Fluorescence to Constitution.* Part II. *Rhodamines of Mellitic Acid.*" By OSWALD SILBERRAD and CHARLES SMART ROY.

Condensation products of mellitic acid with *m*-aminophenol and dimethyl-*m*-aminophenol were described.

The results confirm the conclusions arrived at in the previous paper by one of the authors (Silberrad, *Trans.*, 1906, lxxxix., 1788), in indicating that a quinonoid structure is not an essential integral part of intensely coloured and fluorescent compounds.

The work also appears to show that the lactone ring is much more stable in these compounds than in the less complex phthaleins already known. As in the case of resorcinol compounds (*loc. cit.*), the tetra-aminodixanthyl derivatives exist in two modifications, in which the xanthyl groups are in the meta- and para-positions with regard to one another respectively. The orientation of these groups was determined by the introduction of a third xanthyl group in the case of the meta-compounds.

As both these still contain carboxylic groups in the ortho-position with respect to one another, the formation of trixanthyl derivatives from the meta-compounds only affords an exceptionally good example of steric hindrance.

The following new compounds, together with their salts and derivatives, were described :—

Monoxanthyl derivatives. — 3 : 6-*Diamino*-9-*hydroxy*-*xanthylbenzene*-2-*carboxylactone*-3 : 4 : 5 : 6-*tetracarboxylic acid. Tetramethyl*-3 : 6-*diamino*-9-*hydroxyxanthylbenzene*-*pentacarboxylic acid* and its *pentaethyl* ester. *Tetraethyl*-3 : 6-*diamino*-9-*hydroxyxanthylbenzenepentacarboxylic acid.*

Dixanthyl derivatives—*Octamethyl*-3 : 3′ : 6 : 6′-*tetra*-*amino*-9 : 9′-*dihydroxy*-m-*dixanthylbenzene*-2 : 4 : 5 : 6-*tetracarboxylic acid* and its *ethyl* and *benzyl* esters. *Octaethyl*-3 : 3′ : 6 : 6′-*tetra*-*amino*-9 : 9′-*dihydroxy*-p-*dixanthylbenzene*-2 : 3 : 5 : 6-*tetracarboxylic acid* and its *ethyl* ester. *Tetramethyl*-3 : 6-*diaminotetraethyl*-3′ : 6′-*diamino*-9 : 9′-*dihydroxy*-m-*dixanthylbenzene*-2 : 4 : 5 : 6-*tetracarboxylic acid. Octaethyl*-3 : 3′ : 6 : 6′-*tetra*-*amino*-9 : 9′-*dihydroxy*-p-*dixanthylbenzene*-2 : 3 : 5 : 6-*tetracarboxylic acid.*

Trixanthyl derivatives. — *Hydrated dodecamethyl*-3 : 3′ : 3″ : 6 : 6′ : 6″-*hexa*-*amino*-9 : 9′ : 9″-*trihydroxy*-s-*trixanthylbenzene*-2 : 4 : 6-*tricarboxylactone* and its *triethyl* ester.

DISCUSSION.

Prof. GREEN expressed the opinion that, although the case was more complicated, the facts described by Dr. Silberrad were not greatly different from those which held for the simple rhodamines, and the evidence for or against a quinonoid constitution was of about the same character. The non-formation of a potassium salt was also true of the simple rhodamines, whilst the resistance to esterification might be attributed to steric hindrance, owing to the carboxylic groups being placed between xanthyl residues.

Dr. SILBERRAD, in reply, considered that the action of alkalis on the trixanthyl derivatives, and their insolubility even in concentrated potassium hydroxide, must be regarded as strong evidence in favour of the lactonic formula. The mono- and di-xanthyl compounds, which were shown to be inner salts, possessed the properties of strong tetra- and penta-basic acids respectively.

As to the observation that the rhodamines also formed potassium salts only with the utmost difficulty, it seemed to him that this should be regarded as evidence of their lactonic structure rather than as indicative that the trixanthyl derivatives of mellitic acid, which showed the same lactone-like property, possessed a quinonoid structure.

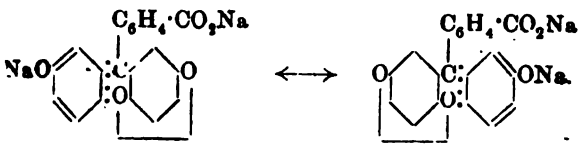
As to the possibility of steric hindrance being the explanation of the difficulty experienced in the esterification of the trixanthyl compounds, here again he thought that the evidence was in favour of the lactonic formula rather than the assumption of any such explanation, for if this were so, the formation of a tetraethyl ester from the meta-dixanthyl derivatives would offer more difficulty than the esterification of the para-compounds; experience showed, however, the converse to be the case, the meta-compound being the more readily esterified.

*200. "*Constitution of the Salts of the Phthaleins and the Cause of Colour in the Triphenylmethane Series.*" By ARTHUR GEORGE GREEN.

Whilst strong evidence has been adduced in favour of the quinonoid structure of the coloured salts and ethers of the phthaleins, there still remain several facts which are not fully explained. One of these is the remarkable difference in colour between the quinonoid diethyl ethers and the alkali salts (orange in the first case and violet-red in the second), whilst another is the circumstance that the substitution of one only of the phenolic hydroxyl groups by methoxyl, in either phenolphthalein or in quinolphthalein, suffices entirely to prevent the formation of coloured salts with alkalis.

By an extension of von Baeyer's theory of oscillatory quinonoid bonds, employing the usual Fittig formula for quinonoid compounds instead of the Graebe formula used by von Baeyer, and by the assumption that the movement of the metallic or acid ion between the two salt-forming groups is the determining cause of the quinonoid oscillation, a satisfactory explanation can be given of the above and other facts, such as the effect of salt formation on colour intensity, the function of auxochromic groups, and the increase of basicity according to the "power law." An explanation is also afforded of the observation that the behaviour of dimethylanilinephthalein, the basic analogue of phenolphthalein, towards acids is analogous to that of the latter towards alkalis, these compounds giving under specific conditions a deep violet colour readily destroyed by an excess of the reagent.

In the quinolphthalein series, the possibility of a meta-quinonoid structure for the coloured alkali salts as suggested by R. Meyer was discussed in relation to the recent work of Green and King. The conclusion was drawn that the facts are best explained by the assumption of an ortho-quinonoid linking oscillating between the two benzene nuclei :—



DISCUSSION.

Dr. SILBERRAD suggested that it would be a fuller and more complete explanation of the experimental evidence if colour were wholly attributed to some inner molecular oscillation, such as that so well illustrated by the examples considered by Prof. Green, and not to the presence of a quinonoid group at all; thus the ethers of these compounds were almost colourless, yet they still contained the same quinonoid constitution, and differed from the parent compound only in that they could not exhibit inner molecular oscillation.

He agreed that the presence or absence of a quinone group exercised considerable influence on the colour of any compound, but so would any other considerable constitutional change.

Further, he thought that the wide application of the quinonoid configuration to coloured compounds by analogy was open to criticism. Prof. Green's observation, that the coloured salts of the rhodamines become colourless in the presence of an excess of acid, did not apply to their mellitic analogues, which yielded coloured solutions with any excess of acid.

Dr. MORGAN remarked that R. Meyer and Desamari's recent demonstration of the existence of tribromo-*m*-benzo-quinone,—

afforded important experimental evidence in favour of the view that certain phthaleins might have a meta-quinonoid structure.

In connexion with the theory that the colour of triphenylmethane dyes was associated with an oscillation of the quinonoid phase, it was noteworthy that diphenylnaphthylmethane dyes could be obtained from naphthastyril, a peri- or 1 ; 8-derivative of naphthalene, because, owing to the configuration of the 1 : 8-naphthylene residue present in these colouring matters, it was difficult to see how this oscillation would take place.

*201. "*The Relation between Absorption Spectra and Chemical Constitution.* Part X. *Unsaturated Acids of the Benzene Series.*" By EDWARD CHARLES CYRIL BALY and KONRAD SCHAEFER.

The absorption spectra of cinnamylideneacetic, cinnamylidenemalonic, benzylidenemalonic, and cinnamic acids have been examined with a view to determining the influence of substitution of the acidic hydrogen atoms by metals and alkyl groups. In the case of cinnamylideneacetone, $C_6H_5{\cdot}CH{:}CH{\cdot}CH{:}CH{\cdot}C(=O){\cdot}CH_3$, a strongly marked absorption band is developed, owing to the isorropesis arising from the conjugation of the carbonyl group with the two double linkings and the phenyl group. In cinnamylideneacetic acid, where the methyl group is replaced by hydroxyl, the absorption band is shifted towards the violet, owing to the fact that the free residual affinity of the carbonyl group is decreased by the juxtaposition of the hydroxyl group. This decrease of free residual affinity is most marked in the case of the readily ionised sodium salt, and least marked with the acid when the ionisation is restrained by the addition of hydrogen chloride. This effect has been observed in the four acids mentioned, and is especially marked in the case of cinnamylidenemalonic acid. The addition of alkali to this acid causes a shift of the absorption band from just outside the visible region to just beyond it, so that the free acid appears to be coloured yellow, and its sodium salt appears to be colourless. The results throw some light on the catalytic action of mineral acids on the esterification process.

*202. "*Relation between Chemical Constitution and Physiological Action in certain Substituted Aminoalkyl Esters.*" By FRANK LEE PYMAN.

Several substituted aminoalkyl esters have been prepared with the object of producing a cocaine substitute ; none of these, however, are suitable for the purpose, since such as possess anæsthetic properties are also toxic and irritant. These substances were prepared by benzoylating (in two cases, phthaloylating) the corresponding amino-alcohols, two of which 1 : 4-*diethanolpiperazine* (m. p. 134—135°) and *β-hydroxy-β-3 : 4-methylenedioxyphenylethyldimethylamine*, have not previously been described. (Where melting-points are not given, the free base did not crystallise). The chemical, physical, and physiological properties of the following aminoalkyl esters and their salts have been examined : — *Dibenzoyloxymethyldiethylamine, dibenzoyloxytriethylamine, βγ-dibenzoyloxydimethylpropylamine, βγ-dibenzoyloxydiethylpropylamine, βγ-dibenzoyloxypiperidylpropane* (m. p. 64—65°), *piperidylethyl benzoate, s-di-β-benzoyloxy-1 : 4-diethylpiperazine* (m. p. 104—105°), *diethylaminoethyl phthalate, piperidylethyl phthalate, β-benzoyloxy-β-3 : 4-methylenedioxyphenylethyldimethylamine.*

203. "*The Passage of Hydrogen through a Palladium Septum, and the Pressure which it Produces.*" By DEMETRIUS TSAKALOTOS.

It was shown by Sir William Ramsay (*Phil. Mag.*, 1894, [5], xxxviii., 206) that hydrogen when it passes through the walls of a palladium tube filled with a gas (N_2, CO, CO_2, CN) at atmospheric pressure, raises the pressure in the tube by nearly one atmosphere when the external pressure of the hydrogen is one atmosphere. There is always a small deficit in pressure, however, for the rise in pressure produced by the entry of the hydrogen never attains that of the atmosphere. The amount of this deficit depends on the nature of the gas originally in the tube.

The experiments to be recorded were made with the object of ascertaining whether, if the tube were initially filled with an indifferent gas, the final pressure would be the theoretical one. Argon was chosen for this purpose. The method employed differed little from that described by Ramsay. The tube was filled with pure dry argon at atmospheric pressure, and at a temperature of 280° (boiling-point of bromonaphthalene) ; it was then exposed on its external surface to an atmosphere of hydrogen, carefully purified and dried. If p be the external pressure of the hydrogen, and p' the internal pressure, that is, the pressure due to the hydrogen after the gas had ceased to enter, the results of the experiments are as follow :—

	I.	II.	III.	Theory.
p'/p . .	0·9281	0·9189	0·9242	1·000

The mean mean result is 0·9238.

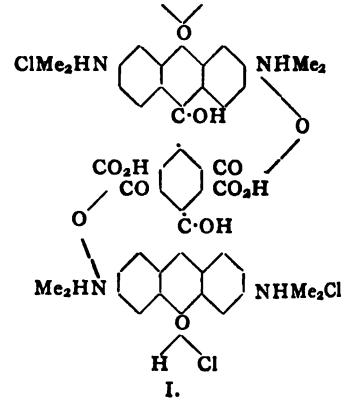
This result does not appreciably differ from that previously obtained by Ramsay ; the figures varied between 0·8984 and 0·9693.

204. "*Constitution of the Phthaleins of Mellitic and Pyromellitic Acids.*" By OSWALD SILBERRAD.

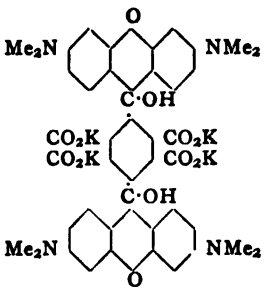
In a previous paper, the author described (*Trans.*, 1906, lxxxix., 1789) condensation products of mellitic and pyromellitic acids with resorcinol to which were assigned carbinol formulæ, as being the most in accord with observed facts. Subsequently, quinonoid formulæ have been suggested (compare, Green, *Proc.*, 1907, xxiii., 12).

In considering the possibility of so formulating these compounds, it should not be lost sight of that the necessity for attributing quadrivalency to the pyrone oxygen atom is hardly in accord with the properties of the compounds in question ; they show none of the basic properties generally attributed to this element when so linked ; on the contrary, they form salts up to the full basicity of the acid groups present. Further, recent work by the author has shown that when present in analogous compounds a quadrivalent oxygen atom impresses a strongly basic influence on the specific properties of the compound.

Thus it has been shown that octamethyltetra-aminodixanthylbenzenetetracarboxylic acid can behave as a base, forming a tetrahydrochloride (I.), or as an acid, forming a tetrapotassium salt (II.), according as the pyrone oxygen is quadrivalent or not.



I.



II.

205. "*A New Form of Gas Burette.*" By ARTHUR EDWIN HILL.

A novel form of gas burette was described which consists of a series of ten bulbs, each with a capacity of 10 cc., connected by short, narrow, graduated tubes. Fractional parts of 10 cc. are measured in a narrow side tube, which has a capacity of 10 cc. graduated into twentieths of a cc.; this is connected to the main vessel by a capillary tube, and forms one arm of a U-shaped manometer, which indicates the pressure of the gas inside the burette. The apparatus is provided with a capillary tap at each end, and is connected by flexible rubber tube with a levelling reservoir.

206. "*A Molecular Compound of Trinitroacetylaminophenol and β-Naphthol.*" By RAPHAEL MELDOLA and JAMES GORDON HAY.

The 2 : 3 : 5-trinitro-4-acetylaminophenol described by one of the authors in a former paper (*Trans.*, 1906, lxxxix., 1935) combines readily with β-naphthol when solutions containing equimolecular weights of the two compounds are mixed together and boiled for a few minutes. Acetic acid or alcohol can be used as a solvent. If the solution is not too dilute, the molecular compound separates out on cooling in dense red prisms, melting with decomposition at 184—185° :—

0·1196 gave 0·2216 CO_2 and 0·0392 H_2O. C = 50·33; H = 3·6.

0·0895 gave 9·9 cc. N_2 (moist) at 14·5° and 755·8 mm. N = 12·89.

0·1245 gave 13·5 cc. N_2 (moist) at 10° and 752·3 mm. N = 12·85.

$C_8H_6O_8N_4, C_{10}H_8O$ requires C = 50·2; H = 3·23; N = 13·02 per cent.

That the compound is "molecular" is shown by its behaviour towards reagents. By alkali alone it is dissolved with an orange colour, which gradually disappears on standing, and, on acidifying, the solution yields only the products of decomposition of trinitroacetylaminophenol and β-naphthol. With alkali and dimethyl sulphate, the compound gives β-naphthyl methyl ether and the products of decomposition of the trinitroacetylaminophenol. By continued crystallisation from acetic acid, the compound is gradually resolved into its components.

The formation of this molecular compound is of interest as, so far, β-naphthol is the only phenol which has been found to enter into combination with the trinitro-compound. The latter, unlike picric acid and other polynitro-compounds, does not appear to form molecular compounds with hydrocarbons. This point is, however, still undergoing investigation, and the authors communicate this note as a preliminary statement of the results of their experiments.

207. "*The Products of Reduction of Azoxybenzene.*" (Preliminary Note). By LESLIE HAMILTON BERRY.

The author is unable to confirm the statement of Schmidt and Schultz (*Ber.*, 1879, xii., 484), that stannous chloride and hydrochloric acid reduce azoxybenzene to aniline, very little benzidine being formed.

He finds, on the contrary, that in no case is the yield of aniline more than 35 per cent, and the principal product is benzidine.

208. "*The Chlorination of Para-nitroaniline.*" By BERNARD FLÜRSCHEIM.

Hitherto no satisfactory method for introducing two atoms of chlorine into *p*-nitroaniline has been known. More especially by the action of chlorine gas much resinous matter was formed. The author finds, however, that a quantitative yield of pure dichloro-*p*-nitroaniline is obtained when *p*-nitroaniline is chlorinated in a mixture of acetic and mineral acids at a low temperature; this result is in accordance with theoretical deduction.

209. "*Syntheses with the Aid of Monochloromethyl Ether.* Part I. *The Action of Monochloromethyl Ether on the Sodium Derivatives of Ethyl Malonate and Ethyl Isopropylmalonate.*" By JOHN LIONEL SIMONSEN.

Monochloromethyl ether and ethyl sodiomalonate readily condense with the formation of *ethyl β-methoxymethylmalonate* :—

$$MeO{\cdot}CH_2Cl + CHNa(CO_2Et)_2 \rightarrow MeO{\cdot}CH_2{\cdot}CH(CO_2Et)_2;$$

this is a colourless oil, which boils at 121—122°/15 mm. When hydrolysed with hydrochloric acid, it is converted into *cis-cyclo*butane-1 : 3-dicarboxylic acid. Other derivatives of ethyl β-methoxymethylmalonate have also been prepared.

When monochloromethyl ether and ethyl *iso*propylsodiomalonate are condensed, *ethyl β-methoxymethylisopropylmalonate* is formed :—

$$MeO{\cdot}CH_2Cl + CNa(CHMe_2)(CO_2Et)_2 \rightarrow MeO{\cdot}CH_2{\cdot}C(CHMe_2)(CO_2Et)_2.$$

This substance boils at 130°—132°/20 mm., and on hydrolysis yields *β-methoxymethylisopropylmalonic acid*, which crystallises in plates melting at 83—84°.

210. "*The Effect of Constitution on the Rotatory Power of Optically Active Nitrogen Compounds.* (Part III.). By REGINALD WILLIAM EVERATT and HUMPHREY OWEN JONES.

An account was given of the resolution of *p*-tolylbenzylmethylallylammonium hydrogen tartrate. The corresponding iodide has been prepared by Wedekind (1903), who found that the camphorsulphonate could not be resolved. This compound is of interest, since it contains two groups, benzyl and tolyl, which have the same mass. The salt of the lævorotatory base was isolated by crystallising about thirteen times from alcohol and ether.

The molecular rotatory power of the ion at 15° is −246°, that of the iodide in alcohol is −257°, and in chloroform is −267°.

The rotatory power of the ion is greater than that of the ion of the corresponding phenyl (167°) or *p*-bromophenyl compound (191°). This is not in agreement with the requirements of Guye's hypothesis as applied to the case of nitrogen, and shows that the constitution of a group may exert such a marked effect on the rotatory power of a compound as to veil completely the effect of mass.

211. "*Studies on the Cobaltinitrites.*" By MARY CUNNINGHAM and FREDERICK MOLLWO PERKIN.

On adding a solution of a salt of a primary amine to a strong solution of sodium cobaltinitrite, a yellow or orange precipitate is produced, which is frequently crystalline. If the solution is not kept cold, the product is apt to decompose. Analysis shows that three molecules of the amine combine with the cobaltinitrite residue. Thus the compound obtained from methylamine has the formula $(CH_3{\cdot}NH_3)_3Co(NO_2)6$. The authors have prepared the methylamine, ethylamine, aniline, *m*- and *p*-bromoaniline, and the *p*-toluidine compounds, all of which have been analysed. It is found that, unless carefully dried as soon as prepared, the compounds are extremely unstable, and even then it is necessary to analyse them very soon after preparation. Diethylamine also forms a crystalline compound, $[(C_2H_5)_2NH_2]_3Co(NO_2)6$, which is fairly stable. The methylaniline compound decomposes before it can be

dried, and apparently a nitroso-compound is produced. From dimethylaniline, the *p*-nitroso-derivative is at once formed.

212. "*The Relation between Viscosity and Chemical Constitution.* Part II. *The Existence of Racemic Compounds in the Liquid State.*" By Albert Ernest Dunstan and Ferdinand Bernard Thole.

The authors have applied the viscosimetric method to the above problem, and have determined the viscosities at 25° of the active and inactive forms of tartaric acid, malic acid, pinene, camphene, and limonene.

They find that, in general, the inactive form has the lower viscosity, and argue that this points to the existence as such of the racemic compound in the liquid state.

They have further examined the viscosity of solutions of sodium ammonium tartrate at different temperatures, and find that a change of curvature appears at 28·7°; hence a new method of determining transition points is to be added to those already in vogue.

213. "*The Formation of Polyiodides in Nitrobenzene Solution.* Part IV. *The Electrolytic Dissociation of the Polyiodides of the Alkali Metals and Ammonium Radicles.*" By Harry Medforth Dawson and Colin Gyrth Jackson.

The authors have investigated the electrolytic dissociation of the polyiodides in nitrobenzene solution by means of freezing-point and electrical conductivity measurements. The data obtained show that the polyiodides are ionised to a considerable extent.

In the case of the polyiodides of potassium, for which several series of freezing-point measurements were made, the depressions, calculated from the chemical dissociation on the assumption that the higher polyiodides are ionised to the same extent as the tri-iodide, are in good agreement with the observed values. It is probable that the penta-, hepta-, and ennea-iodide of each alkali metal and ammonium base are equally ionised, although the degree of ionisation appears to vary with the nature of the cation. On the other hand, evidence has been obtained that, in some cases, the tri-iodides are not ionised to the same extent as the higher polyiodides.

As the result of comparative measurements of the conductivity of solutions of the iodides and tri-iodides it is found that the tri-iodides are electrolytically dissociated to a much greater extent than the iodides. In the case of tetrapropylammonium iodide, for which, on account of its solubility in nitrobenzene, a comparison by the freezing-point method has been possible, the conclusions drawn from the conductivity data are completely confirmed.

The mobilities of the polyiodide ions have been deduced by combining the results of transport measurements with the freezing-point and conductivity data.

Finally, determinations of the molecular solution-volume of iodine and the polyiodides have been made. The results show that the volume of combined iodine differs from that of iodine in the free condition.

214. "*The Direct Action of Radium on Copper and Gold.*" By Edgar Philip Perman.

The direct action of the radiations from a radium salt on a pure copper salt has been investigated; the salts were placed in small capsules facing one another in a wide tube, which was exhausted and sealed.

In the case of gold, the two salts were left in solution together, and in another experiment, gold and radium barium sulphate were precipitated together, thoroughly washed, and allowed to stand.

After four months, no trace of lithium could be detected by spectroscopic examination, and the conclusion is drawn that if any copper or gold is converted into lithium, it cannot be more than the one hundred-millionth part of the weight of the radium salt per day in the case of copper, and the one two-hundred-millionth part in the case of gold, under the conditions of the respective experiments.

215. "*The Atomic Volumes of Phosphorus.* Part II. *Phosphorus and Bromine.*" By Edmund Brydges Rudhall Prideaux.

The specific volumes of liquid phosphorus pentabromide and the pressures of its saturated vapour at various temperatures have been measured. From the values of the vapour pressures from 20° to 100°, and of the coefficients of expansion and molecular volumes from 85° to 100° and 100° to 165°, the true boiling-point is placed at 106°, and the molecular volume at this temperature is 157·3. A review of the molecular volumes of the chlorides, bromides, phosphoryl chloride, and phosphoryl dichlorobromide shows that the atomic volumes of tervalent are greater than those of quinquivalent phosphorus.

216. "*Isomeric Chromous Chlorides.*" By Elizabeth Mary Rich.

The author has devised a rapid and convenient method of preparing pure chromous chloride, which is thus rendered practically available for use in all kinds of gas analysis. Two isomeric series of hydrates and their solutions have been studied. The green isomeride has the formula $CrCl_2,4H_2O$, identical with the blue salt. Its green solution contains the ions CrCl· and Cl′.

217. "*The Relative Atomic Weights of Hydrogen and Chlorine.*" By Robert Whytelaw Gray and Frank Playfair Burt.

The ratio H : Cl has been measured by a method which is different from that employed by Dixon and Edgar (*Phil. Trans.*, 1905, ccv., 169), or by Noyes and Weber (*Journ. Am. Chem. Soc.*, 1908, xxx., 13). The investigation was carried out with hydrogen chloride obtained from several sources, and purified by fractional distillation. (1). The density of the gas was determined, the gas being condensed on charcoal and weighed. The mean of twenty experiments gave for the weight of a normal litre of gas the value 1·63900 ± 0·00004 grms., or making a small correction for the amount of gas condensed on the surface of the walls of the density bulb and absorbed by the tap grease, 1·63885 grms. (2). The gas was analysed volumetrically by passage over heated aluminium. By this means the volume of hydrogen contained in exactly two volumes of hydrogen chloride measured at 0° and 760 mm. was determined, and this gave the exact ratio of the volumes of hydrogen and hydrogen chloride which under standard conditions contain the same number of molecules.

Eight measurements with gas from different sources gave for the volume ratio H_2 : HCl the mean value 1·00790 ± 0·00002.

From these two results, the molecular weight of hydrogen chloride may be directly calculated as follows:—

If D_{HCl} represents the measured weight of a litre of hydrogen chloride at 0° and 760 mm., lat. 45°, and D_{H_2} represents the known weight of a litre of hydrogen at 0° and 760 mm., lat. 45°, and if—

$$\frac{V_{H_2}}{V_{HCl}}$$

expresses the ratio of the volumes of hydrogen and hydrogen chloride, which at 0° and 760 mm. contain the same number of molecules; then the molecular weight of hydrogen chloride is given by the expression—

$$\left(\frac{D_{HCl}}{D_{H_2}} \times \frac{V_{HCl}}{V_{H_2}}\right) \times 2.$$

On the basis of Morley's values for the normal density of hydrogen, namely, 0·089873 grm. per litre, and for the atomic weight of hydrogen, namely, 1·00762, the molecular weight of hydrogen chloride is—

$$\left(\frac{1·63885}{0·089873} \times \frac{2}{1·00790}\right) 1·00762 = 36·461,$$

and the atomic weight of chlorine = 35·453. This agrees almost exactly with the results of Noyes and Weber, namely, Cl = 35·452, but is 1/3500 part lower than Dixon and Edgar's number, namely, 35·462.

218. "*The Interaction of Hydrogen Dioxide and Sulphides.*" By Maud Gazdar and Samuel Smiles.

The usual method of preparing sulphoxides from sul-

phides by means of concentrated nitric acid is useless when applied to sensitive compounds, such as di-*p*-aminophenyl sulphide or thiodiglycollic acid. Hydrogen dioxide in acetone or aqueous solution reacts at the ordinary temperature with the sulphides, giving almost quantitative yields of the corresponding sulphoxide. The reaction can be applied to sulphides of very varied character, for, besides amyl, benzyl, and phenetyl sulphoxides, the hitherto unknown *di-p-aminophenyl sulphoxide* and *thionyldiglycollic acid* were obtained. Diphenylene disulphide and tetranitrodiphenyl disulphide are not attacked by the reagent under ordinary conditions.

219. "*The Influence of Solvents on the Rotation of Optically Active Compounds.* Part XIII. *Ethyl Tartrate in Aromatic Nitro-derivatives. Influence of Temperature change on Rotation in Solution.*" By THOMAS STEWART PATTERSON.

The influence of the aromatic nitro-derivatives as solvents on the rotation of ethyl tartrate was described. The effect of temperature-change on the rotation of these and other solutions was also discussed.

220. "*The Isomeric Menthols.*" (Preliminary Note). By ROBERT HOWSON PICKARD and WILLIAM OSWALD LITTLEBURY.

According to theory there should be eight optically active forms of 1-methyl-4-*iso*propyl*cyclo*hexan-3-ol corresponding in pairs with four racemic compounds. Of these menthols, one, the ordinary *l*-menthol, occurs in nature, whilst the existence of others has been indicated by the researches of Beckmann, Kondakoff, and Brunel. It is now found that these compounds can be separated by the fractional crystallisation of suitable esters, such as the hydrogen phthalates, hydrogen succinates, &c.

By the reduction of thymol with hydrogen in the presence of reduced nickel, Brunel (*Comptes Rendus*, 1905, cxl., 252) obtained a low-melting thymomenthol, which he considered was transformed into an isomeride by conversion into an ester and subsequent hydrolysis. The authors have been able to show that this "α" thymomenthol of Brunel contains three (or perhaps all four) of the racemic compounds mentioned above. Up to the present, two of these have been fully characterised and resolved into their optically active components by fractional crystallisation of various alkaloidal salts of some of their acid esters (compare Pickard and Littlebury, *Trans.*, 1907, xci., 1974).

The first of these racemic menthols melts at 25°, and forms a *hydrogen succinate*, m. p. 86°, and also a *hydrogen phthalate*, m. p. 131°, the latter of which by fractional crystallisation of either its cinchonine or brucine salt yields a lævorotatory isomeride. When hydrolysed, this lævo-ester yields a menthol identical in melting-point and rotation with the natural menthol.

The second of the racemates melts at 48—50°, and forms a *hydrogen phthalate*, m. p. 175—177°, which is readily resolved into its active components by crystallisation of the brucine salt.

SOCIETY OF CHEMICAL INDUSTRY.
(LONDON SECTION).

Ordinary Meeting, November 2nd, 1908.

Dr. LEWKOWITSCH in the Chair.

A PAPER entitled "*Chemical Industry in relation to Agriculture*," by Prof. ADOLF FRANK (Charlottenburg), was read by the Chairman.

After referring to the great services of Liebig to agriculture, the author gave an historical survey of the manufacture and agricultural uses of phosphates and the production of potash. Ammonium sulphate, Chile saltpetre, and the utilisation of atmospheric nitrogen were also discussed, and an account was given of the author's own work in effecting the combination of atmospheric nitrogen with carbides of the alkalis and the alkaline earths. By decomposing the calcium cyanamide with water under high pressure ammonium salts are formed. Possibilities of calcium cyanamide as a fertiliser are dealt with, and some statistics relating to the output of calcium carbide are included. The work of Mond on the simultaneous production of power gas has made it possible to utilise the large stores of energy accumulated in bog areas in the form of peat. The author and Caro, with the assistance of Mond, have been able without difficulty to gasify peat containing 50 to 55 per cent of water.

NOTICES OF BOOKS.

Dyeing and Cleaning. By FRANK J. FARRELL, M.Sc. (Vict.). London: Charles Griffin and Co., Ltd. 1908.

THIS book contains complete accounts of the general principles of the usual methods of dry and wet cleaning, and includes a chapter on dyeing which is written specially from the point of view of the job dyer, so that the problems which he has to face are treated at greater length than would be necessary for a manual on the dyeing of piece goods. The book is thoroughly practical, and will be a useful handbook for dyers and cleaners. A short chapter on technology gives brief accounts of the origin of the different animal and vegetable fibres, and the action of acids and alkalis upon them is described in some detail. Dry cleaning by various processes is next discussed, the treatment of the articles to be cleaned being described from the moment they are received and marked, while a chapter at the end of the book explains methods of finishing, which are of more importance for wet-cleaned and dyed than for dry-cleaned articles. Wet cleaning and dyeing are treated in outline, though no doubt fully enough for the purpose for which the book is intended, and special methods which have to be applied to skins, furs, feathers, &c., are given some consideration.

From the Interim Report issued by the Departmental Committee on Dangerous Trades the sections relating to dry cleaning are reprinted.

A defect of the work, which is not uncommon in this kind of book, is the reproduction of pictures of various types of machines. These often occupy the whole page, except perhaps for a few lines top and bottom; they are of no particular use to the practical man, who is probably already well acquainted with the machines, nor to the beginner, who learns far more from the examination of one or two models, and who moreover would want to study sections more than photographs of exteriors; thus they add to the bulk of the book without increasing its value in the same proportion.

The Extra Pharmacopœia of Martindale and Westcott. Revised by W. HARRISON MARTINDALE, Ph.D., F.C.S., and W. WYNN WESTCOTT, M.B.Lond., D.P.H. Thirteenth Edition. London: H. K. Lewis. 1908.

THE thirteenth edition of the Extra Pharmacopœia contains information relating to many new remedies and fresh preparations which have recently come into use, and the developments of various branches of pharmacy are summarised. The valuable work which has been done during the last few years on vaccines and antitoxins is reviewed, and several new vaccines are described. The section on radiology has been brought up to date, and many parts of the analytical section have been reprinted; for the first time a list of the results of the analysis of various patent medicines is included. In spite of the fact that the new edition contains much more material than the old, the book is less thick than before, owing to the use of smaller type and thinner paper.

House Painting. By ALVAH HORTON SABIN, M.S. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1908.

THE householder who likes to have some idea of what he is paying for when he has his house painted may refer to this book for practical information on suitable pigments, methods of applying paints, &c. The painter will probably find nothing in it that he did not know before, though it contains many counsels of perfection which he finds it convenient to forget. Glazing and papering are included, and copious directions are given to help the amateur to do his own papering and decorating.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

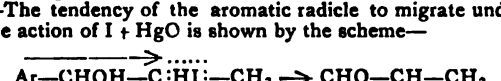
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 16, October 19, 1908.

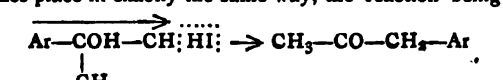
Precipitated Silica.—Henry Le Chatelier.—Silica loses all its water at 100° under atmospheric pressure, or in a vacuum at the ordinary temperature. Hence its hydrates, if they exist, are not stable. It is known that a hydrate at temperatures at which it is stable has a tension of efflorescence lower than the vapour tension of pure water, but this vapour tension increases more rapidly than that of water, and at a certain temperature, which is generally lower the less stable the hydrate is, the substance and water have exactly the same vapour tension. Above this temperature the hydrate is not stable. The temperature is 32° for sodium sulphate and 150° for calcium sulphate. Gelatinous silica, on the other hand, keeps the same appearance and consistency even after it is heated to 320°, whereas the destruction of a hydrate would be detected by a change in the appearance of the material. The only plausible explanation is the assumption that silica does not form hydrates, but always exists in the anhydrous state. Precipitated silica is in a state of fine division, and forms pastes of variable consistency with water. It would readily pass through filter-paper, although insoluble in water.

New Method of Opening up Ferro-alloys, particularly Ferro-Silicons.—Paul Nicolardot.—If coarsely powdered ferro-silicon is heated with sulphur chloride bubbles of gas are evolved at 70°; the action becomes violent at 100°, but then white fumes are evolved, indicating the formation of silicon chloride. With ferro-chromium the action does not begin till a temperature above 120° is reached, while with ferro-titanium it begins at 70°. If the vessel in which the reaction takes place is fitted with a cork through which the tube of a reflux condenser passes, all the chlorides can be retained, and thus the refractory alloys are completely opened up. The same method can be employed for analytical purposes for ferro-titaniums and ferro-silicons, the titanic oxide and silicon being weighed.

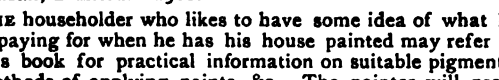
Migration of Naphthyl Group in Iodohydrins of the Naphthalene Series.—MM. Tiffeneau and Daudel.—The tendency of the aromatic radicle to migrate under the action of I + HgO is shown by the scheme—



In the naphthalene derivatives, *e.g.*, the allyl, isoallyl, pseudoallyl, and vinyl compounds the phenyl migration takes place in exactly the same way, the reaction being—



By analogy from the reaction with styrolene it must be assumed that the reaction with naphthylethylene is accompanied by the migration of a naphthyl group—



Modification of the Method of Preparing Monomethylamine by Brominated Acetamide.—Maurice François.—The brominated solution is prepared by dissolving acetamide in bromine, adding water and calcium carbonate, and filtering the liquid. Thus a clear red liquid is obtained It is added to cold caustic soda solution and the white turbid liquid is heated in a special tube to 100°; the raw product is distilled in a current of steam and the monomethylamine can then be converted into chlorhydrate by the action of hydrochloric acid. The yield is 72 per cent of the theoretical yield.

State of Colouring Matters in Solution.—L. Pelet-Jolivet and A. Wild.—The authors have studied different coloured solutions, *e.g.*, fuchsine, methylene blue, &c., by the method of conductivity and with the aid of the ultramicroscope, and find that the colouring matter exists in the dissociated state, or in some cases in the colloidal state. The addition of electrolytes by the action of ions of opposite signs favours the passage of the coloured solution into the state of a false solution. Colouring matters in solution and dissociated form ions of unequal dimensions; the inorganic ion Cl, K, Na, &c., is always relatively smaller than the organic ion.

MISCELLANEOUS.

Royal Institution.—The eighty-third Christmas course of juvenile lectures, founded at the Royal Institution in 1826 by Michael Faraday, will be delivered this year by Prof. William Stirling, M.D., LL.D., D.Sc., his subject being "The Wheel of Life." The course, which will be fully illustrated, commences on Tuesday, December 29, at 3 o'clock, and will be continued on December 31, 1908, January 2, 5, 7, and 9, 1909.

Chemical Society Research Fund.—A meeting of the Research Fund Committee will be held in December next. Applications for grants, to be made on forms which can be obtained from the Assistant Secretary, must be received on or before Monday, December 7, 1908. All persons who received grants in December, 1907, or in December of any previous year, whose accounts have not been declared closed by the Council, are reminded that reports must be in the hands of the Hon. Secretaries not later than Tuesday, December 1. The Council wish to draw special attention to the fact that the income arising from the donation of the Worshipful Company of Goldsmiths is to be more or less especially devoted to the encouragement of research in Inorganic and Metallurgical Chemistry. Furthermore, that the income due to the sum accruing from the Perkin Memorial Fund is to be applied to investigations relating to problems connected with the Coal-tar and Allied Industries.

MEETINGS FOR THE WEEK.

MONDAY, 23rd.—Royal Society of Arts, 8. (Cantor Lecture). "Twenty Years Progress in Explosives," by O. Guttmann.

WEDNESDAY, 25th.—Royal Society of Arts, 8. "The Goldfields of Eastern Peru and Bolivia," by Sir W. Martin Conway.

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To ensure insertion in next issue, Advertisements should be addressed to THE MANAGER, *and must reach the Office not later than* 10 *a.m. on* WEDNESDAY.

CHEMICAL SOCIETY RESEARCH FUND.

A Meeting of the Research Fund Committee will be held in DECEMBER, 1908. Applications for Grants, to be made on forms which can be obtained from the Assistant Secretary of the Society, Burlington House, W., must be received on or before MONDAY, DECEMBER 7th.

The Council wish to draw special attention to the fact that the income arising from the donation of the Worshipful Company of Goldsmiths is to be more or less especially devoted to the encouragement of research in Inorganic and Metallurgical Chemistry. Furthermore, that the income due to the sum accruing from the Perkin Memorial Fund is to be applied to investigations relating to problems connected with the Coal-tar and Allied Industries.

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THE CHEMICAL NEWS.

Vol. XCVIII., No. 2557.

THE PRESENT STATE OF OUR KNOWLEDGE OF RADIO-ACTIVITY.*

By A. DEBIERNE.

(Concluded from p. 248).

V.

The following is the principal fact which has led to these theories:—In different circumstances it is possible to produce, by the aid of radio-active substances of constant and permanent activity, phenomena of radio-activity the intensity of which decreases with time.

Pierre Curie and Mdme. Curie first observed this new property in the salts of radium. They placed in the same receptacle different objects and a certain quantity of a product containing radium; they proved that the objects, after having been thus left beside radium salt, themselves became radio-active; they spontaneously emitted a radiation perfectly analogous to that of radium, producing the ionisation of the air, phosphorescence, and photographic action. These radio-active phenomena disappear fairly quickly with time, after the action of the radium has been stopped. Pierre Curie and Mdme. Curie called them induced radio-activity.

A short time afterwards Rutherford observed the same phenomenon with thorium salts. He proved, moreover, that gases which have passed over thorium oxide possess a radio-activity which rapidly decays with the time. He at once conjectured that the gases contain a special radio-active gaseous substance, to which he gave the name thorium emanation.

I observed the same phenomena with actinium salts. Later, I noticed that the temporary phenomena of radio-activity could be obtained by carrying out certain chemical separations. Thus, if a barium salt is added to a solution of an actinium salt and the two metals are then separated by the action of ammonia, it is found that the barium salt acquires a fairly strong activity which disappears with time. As we shall see later, the presence of the barium has no influence upon the production of the phenomenon.

The phenomenon of induced radio-activity was studied by Pierre Curie and various collaborators. It was shown that the immediate cause of the phenomenon is in the gas which has been in contact with the radium salt; the gas is itself radio-active, and also contains an emanation which disappears with time. The laws of the decrease of the intensity of the different phenomena were also studied in detail. The presence of an emanation was proved in the case of actinium as well.

A general theory which linked up all these phenomena was put forward by Rutherford and Soddy, after a series of experiments carried out with thorium. They first proved that it is possible to obtain with the compounds of thorium a phenomenon analogous to that which I had discovered with actinium; *i.e.*, temporarily radio-active substances could be extracted by the aid of chemical separations. They studied the laws of the phenomenon, and they interpreted it as follows:—They supposed that the existence of radio-active phenomena, whether these phenomena appear to be permanent or temporary, is connected with the presence of special radio-active substances, and that the disappearance or appearance of the phenomena of radio-activity indicates the destruction or formation of these active substances. Thus this is an extension of the views which led to the discovery of radium.

They named these new active substances in different ways. Thus, the hypothetical substance which can be separated chemically from thorium, and the activity of which disappears gradually with time, they called thorium X. They showed that thorium X is produced continuously by thorium, and that its activity is an important part of the activity of thorium salts; it is the thorium X which produces the thorium emanation, and the emanation produces the induced activity. This induced activity, detected in substances which have been in contact with the emanation, is attributed to the deposit of new active substances, which have been called thorium A and thorium B.

Now, the researches of Pierre Curie on the disappearance of the activity excited by radium salts showed that the existence of several different kinds of radio-activity following on one another must be admitted. According to the above theory this shows the presence of several active substances derived from radium emanation; these have been called radium A, radium B, and radium C. The laws of the phenomenon also show that the formation of one term of a series is proportional to the destruction of the preceding term; for instance, the formation of radium C is proportional to the destruction of radium B, which is most simply explained by supposing that radium B is destroyed to form radium C.

Researches on actinium have also shown a succession of different substances, actinium X, emanation, actinium A, actinium B.

In fact, these laws of the formation and destruction of active substances are exactly the same as those observed with ordinary chemical reactions in the case of monomolecular transformations. When a substance A undergoes a monomolecular transformation the quantity which is transformed in a given time is proportional at each instant to the quantity still in existence, $dq - Kqdt$. K is the coefficient of velocity of the transformation. The law of decay is thus a simple exponential law; it has been verified in a certain number of cases, in particular in the case of the transformation of acetochloranilide into *p*-chloracetanilide. The verification is generally very difficult to obtain in the case of ordinary chemical actions, for the determinations are often almost impossible. In the case of the radio-active transformations, on the contrary, it is possible to obtain a perfect verification by measurements of the radio-activity.

If a single active substance was present, and in decaying it gave rise to an inactive substance, the phenomenon would be very simple. The radio-activity would diminish according to a simple exponential law; *i.e.*, the same proportion of activity would disappear in equal times; for example, half of the activity would always take the same time to disappear.

This law is represented by the equation $I = I_0 e^{-\lambda t}$. If the times are taken for abscissæ and the logarithms of the activities for ordinates, a straight line is obtained.

If, on the other hand, the substance A gives rise to another active substance B, and if this produces another substance C, also active, &c., the phenomena become much more complicated. Let us suppose that at first only the substance A exists; it decays according to the above law, and the activity due to its presence will decrease according to a simple exponential. The quantity of the substance B is at first nil; it increases as it is produced by A, and the production is proportional to the destruction of A. But B also disappears, and this disappearance is proportional to the quantity of B; thus the quantity of B, and hence the activity due to its presence, will first increase, then reach a maximum, and finally decrease progressively. It may be shown that the law of the variation of the concentration of the substance B, and hence of its activity, can be represented by the difference of two exponentials, one characteristic of the destruction of A and the other characteristic of the destruction of B. For the variation of C we should get three exponentials, &c.

The total activity at each instant is equal to the sum of the activities, due respectively to the substances A, B, C, &c.; *i.e.*, to the sum of exponentials with coefficients, the value of which depend on the activity of each substance.

* *Revue Générale des Sciences*, 1908, xix., 691.

TABLE I.—*Relations between Radio-active Substances.*

Substances.	Properties.	θ.	T.	Radiation.
Uranium	Atomic weight = 240. Iron group: soluble in ammonium carbonate; nitrate soluble in organic solvents.			α = 3·3 cm.
↓				
Uranium X	Iron group: carried down by $BaSO_4$; nitrate slightly soluble in organic solvents, acetone, ether, &c.	31·6 days	22 days	β
↓				
?				
Thorium..	Atomic weight = 232. Precipitated by ammonia and oxalic acid.			α?
↓				
Mesothorium . ..			5·5 years	β
↓				
Radio-thorium ..	Iron group.	1060 days	740 days	α=3·9 cm.
↓				
Thorium X	Not precipitated by ammonia.	5·2 days	3·6 days	α=5·7 cm.
↓				
Thorium emanation	Inert gas, condensible.	78 secs.	54 secs.	α=5·5 cm.
↓				
Thorium A	Induced active deposit.	15·8 hours	11 hours	β, slow
↓				
Thorium B	Induced active deposit.	1·44 hours	1 hour	α=5·0 cm.
↓				
Thorium C	Induced active deposit.	1·44 hours	Very short	α=8·6 cm. β, γ
↓				
?				
Radium	Atomic weight = 226·5. Spectrum; group of alkaline earths. Continuously produces helium			α=3·5 cm.
↓				
Radium emanation .	Atomic weight by diffusion = about 100? Spectrum? Inert gas, condensible.	5·8 days	3·9 days	α=4·23 cm.
↓				
Radium A	Induced active deposit.	4·3 mins.	3 mins.	α=4·83 cm.
↓				
Radium B	Induced active deposit.	37·4 mins.	26 mins.	Slow β-rays
↓				
Radium C	Induced active deposit separated by electrolysis.	27·4 mins.	19 mins.	α = 7·06 cm. β, γ
↓				
Radium D	One of the constituents of radio-lead; properties very much like those of lead.	57·5 years	40 years?	No rays
↓				
Radium E	Separated from D by electrolysis.	8·6 days	6 days	β
↓				
Radium F or polonium	Sulphide insoluble in acids; precipitated with basic salts of bismuth; precipitated by stannous chloride.	201 days	140 days	α 3·86 cm.
↓				
?				
Actinium	Group of rare earths? Precipitated by oxalic acid and by hydrofluoric acid. Goes with neodymium and samarium in the fractionation of the ceric earths. Continuously produces helium.			No rays
↓				
Radio actinium ..	Separated by electrolysis and by fractionation with ammonia.	28 days	19·5 days	α=4·8 cm.
↓				
Actinium X	Soluble in ammonia.	14·7 days	10·2 days	α=6·55 cm.
↓				
Actinium emanation	Inert gas, condensible. Atomic weight = 70?	5·6 secs.	3·9 secs.	α=5·8 cm.
↓				
Actinium A	Induced active deposit.	50 mins.	36 mins.	No rays
↓				
Actinium B	Induced active deposit. Separated by electrolysis.	2·9 mins.	2 mins.	α=5·5 cm. β, γ
↓				
?				

The law of the variation of activity may thus be very complicated. However, the experimental decay curves have been analysed, and thus the number of active substances derived from one another has been deduced with certainty, and also the exponential constants characteristic of their decay. These decay curves are sometimes simplified; if, for example, the curve is represented by the difference between two exponentials of the form $Ke^{-\lambda t} - K'e^{-\lambda' t}$, where the coefficient λ is decidedly greater than λ', when t is increased the term containing $e^{-\lambda t}$ becomes, finally, very small, and thus the curve is reduced to a simpler exponential characteristic of one of the substances. Deducting the experimental curve of this exponential it is easy to obtain the second exponential. By this method Pierre Curie showed the differences of the exponentials in the case of the induced activity of radium, and thus deduced the elementary law of the phenomenon. Very valuable information can also be obtained by altering the initial conditions.

All these temporarily active substances can be distinguished from one another by means of other properties. In the first place the nature of the radiation; certain substances emit only α-rays, others β-rays, and others a mixture of α-, β-, and γ-rays; finally, certain substances do not emit rays or at most only a very feeble radiation.

Moreover, the researches of Bragg and Kleemann have shown that when a substance emits α-rays these rays are all of one kind; they all have the same range, which implies the same initial velocity of projection. The measurement of this range is comparatively easy, and by it certain active substances can be identified.

Finally, true separations can be effected either by physical or chemical processes. We have already seen that some substances are gaseous and that others are deposited on solid bodies. The gaseous emanations can generally be easily separated, either from solid salts or from solutions. They may even be condensed on solid walls by lowering the temperature sufficiently. Rutherford, who discovered this phenomenon, has shown that in the case of radium the condensation takes place within a small temperature interval; the condensation can be effected at the ordinary temperature on wood charcoal.

The different products which are included in induced radio-activity can also be separated by fractional distillation at a high temperature. Radium B and radium C can thus be nearly completely separated.

We have seen that chemical methods can be used to separate thorium X and actinium X; similar methods can be used to separate the constituents of induced radio-activity. Thus the active deposit of radium can be dissolved in an acid and then electrolysed. Radium C can thus be completely separated from radium B.

Do the chemical separations thus effected correspond exactly to the properties the substances would have if they could be obtained in ponderable quantities? This question is difficult to answer, but it must be admitted that the existence of these processes is in favour of Rutherford and Soddy's hypothesis that the phenomena of temporary activity are due to chemical substances; their existence is admittedly ephemeral, but during their short life they appear to possess well-defined chemical and physical properties.

Each active substance which disappears more or less rapidly may also be characterised by different properties, but it may be identified with certainty by the law of its spontaneous decay, which is determined exactly by the exponent λ; this exponent is the coefficient of velocity of the transformation. We know that in chemical reactions these coefficients of velocity are much influenced by external agents, temperature and pressure, and even by the presence of certain foreign bodies, catalysers. In radio-active transformations the coefficients of velocity seem to be absolutely independent of external conditions. For instance, Pierre Curie has shown that the velocity of the destruction of radium emanation does not vary from the temperature of liquid air to high temperatures.

This is a special characteristic of these phenomena, which is connected with the atomic nature of the property of radio-activity. Just as the nature of the atom and its mass are unaffected by the agents we are dealing with, the radio-active properties are not modified by external actions.

Instead of defining the law of the destruction of an active substance by the coefficient of velocity, it may be defined by a coefficient which has an interesting theoretical signification. If we consider the different atoms of an active substance in course of destruction, certain atoms will undergo the transformation at the end of a very short time; others, on the other hand, will have a much longer life; a mean duration of life of the different atoms can be deduced by employing the law of destruction. The quantity $\theta = 1/\lambda$ has been found for this mean duration of life, and θ is the coefficient generally used.

The law of transformation is very often defined by the interval of time, at the end of which half the substance is transformed; *i.e.*, at the end of which the activity due to the substance has diminished to one-half. These two constants are connected by the equation $T = \theta \log 2$.

For instance, for radium emanation $\theta = 136$ hours, $T = 92$ hours.

Many experiments were necessary to determine the succession of the different active substances derived from the principal substances known.

One of the most interesting results, chiefly due to Rutherford, is the proof that polonium, the activity of which as we have seen decreases slowly with time, may be connected with the radium series, and also that radio-lead is a mixture the different members of which are also derived from radium.

I append a table of different active substances the succession of which has been proved. The table includes four series, derived from uranium, thorium, radium, and actinium. For each substance I have given the interval of time corresponding to the half decay, the mean duration of life, the nature of the rays emitted with their range when it is known, and finally some information as to the physical and chemical properties which may be employed to separate them.

In a second article I shall continue the account of our present knowledge of radio-activity.

PRELIMINARY NOTE ON A NEW ELEMENT ALLIED TO MOLYBDENUM.

By MASATAKA OGAWA, Rigakushi.

Introduction.

IN the course of an examination of molybdenite in connection with my work on Nipponium, a substance, which was supposed to be ammonium vanadate, was obtained and set aside as such (*Journ. Coll. Sci., Imp. Univ.*, xxv., Art. 15, p. 7; CHEMICAL NEWS, xcviii., 249). A further examination of this substance has shown, however, that it is not ammonium vanadate, but that it is a compound of a new element, closely allied to molybdenum, and, indeed, associated with it in this compound; and although the results so far obtained are still very incomplete, they are now published in the hope that they may prove of sufficient interest.

It is possible and even likely that the "new tin-group element in thorianite," an account of which has been recently given by Miss Evans (*Journ. Chem. Soc.*, 1908, xciii., 666), is identical with this element, inasmuch as the latter has been found not only in molybdenite but also in thorianite, and two at least of the very few characteristic reactions ascribed to her element by Miss Evans, namely, the formation of a brown sulphide, insoluble in hydrochloric acid but soluble in hot nitric acid, and its solubility

* *Journal of the College of Science, Imperial University, Tokyo, Japan*, vol. xxv., Article 16.

in ammonium carbonate to form a deep brown solution, are likewise produced by the element now to be described. The formation of a golden-brown syrup on evaporation of a nitric acid solution of the sulphide, which she appears to regard as another characteristic of her element, could, however, in the case of the element forming the subject of this paper, be observed only with the impure sulphide, which, in some form or other, was found to contain a small quantity of iron, besides, perhaps, some other elements. Again, on reducing with hydrogen, the oxide prepared from such sulphide produced a metal, which, in agreement with Miss Evans' observation, was fusible at a bright red heat, but the metal obtained from the purified oxide was found to be non-fusible. It appears, therefore, that, if Miss Evans' element is really identical with mine, I have got it in a much purer state. In any case it is unfortunate that in consequence of her account being much too short no exact comparison can be instituted between the two. It is also unfortunate that, in spite of her positive statement as to the existence of molybdenum in thorianite, she does not tell us how this element has been completely removed, the sulphide of molybdenum also dissolving in ammonium carbonate to form a deep brown solution.

There is one other point which should be noted before going further. Miss Evans says that the sulphide of her element is soluble in water. The solubility of the sulphide upon which I worked may be inferred from the fact that one of its sources was the sulphide residue from thorianite, which had escaped precipitation by hydrogen sulphide (see *prox.*), and also from the fact that a solution of the purified oxide in ammonia was almost completely precipitated by hydrogen sulphide, *only* after acidification and *on boiling*. The presence of the element in the filtrate from the hydrogen sulphide group may therefore be most probably ascribed to the incomplete heating during precipitation by hydrogen sulphide.

Preparation from Molybdenite of a Compound containing the New Element and its Examination.

As the condition under which the supposed ammonium vanadate was previously obtained was such that it did not ensure its separation quantitatively and in the pure state, supposing it to be really ammonium vanadate, the following procedure has been adopted. After igniting in oxygen the powdered mineral was extracted with ammonia. The residue, still containing some unaltered mineral, was repeatedly boiled with concentrated nitric acid, and the whole evaporated to dryness and again extracted with ammonia. The final residue, on digestion with ammonium sulphide, gave a deep reddish brown solution, which was filtered from the insoluble portion containing the sulphides of lead, bismuth (?), iron, nipponium, and zinc, and the hydroxide of aluminium. The dark brown precipitate, obtained on acidifying the solution with hydrochloric acid and heating, was decomposed with nitric acid, and the yellowish brown syrupy residue, left on evaporation of the nitric acid solution, ignited in the air. The dark brown residue thus obtained was fused with potassium nitrate, and the fused mass extracted with water, when a small quantity of a reddish brown substance, consisting chiefly of ferric oxide, was left undissolved. The aqueous alkaline extract was neutralised with nitric acid and, after concentration, mixed with an equal volume of a saturated solution of ammonium chloride, followed by the addition of one-third of the total volume of 90 per cent alcohol. A crystalline precipitate, apparently the same as that which had been previously obtained and set aside as ammonium vanadate, was produced on standing for some time, and this was washed, first with a saturated solution of ammonium chloride and then with dilute alcohol.

Judging from the mode of procedure no other known substance but ammonium vanadate could have come down as a crystalline precipitate under the conditions above detailed, but the reactions described below clearly show that it is not ammonium vanadate. Neither can it be ammonium molybdate, for this salt is not known to be precipitated by ammonium chloride. It is true that the compound gives some of the reactions of molybdenum, which it undoubtedly contains in one form or another, but the fact above alluded to, that it has been obtained under conditions which exclude the possibility of ammonium molybdate being precipitated pure and simple, makes it exceedingly probable either that the compound above obtained is a complex salt containing molybdenum and the new element or that it belongs to the class of mixed crystals.

The yield of the compound was about 7 grms. from 400 grms. of molybdenite. It is remarkable that the existence, in this mineral, of such a comparatively large amount of a new element should have hitherto escaped observation, but this may be explained by the fact that the element is very closely allied to molybdenum and that its equivalent weight does not differ much from that of the latter.

Properties of the Compound.—The compound above obtained, which is evidently an ammonium salt, crystallises in minute prisms, easily soluble in water, and its aqueous solution gave the following reactions :—

(1) Hydrogen sulphide, in presence of hydrochloric acid, only gave a brown coloration without producing any precipitate, but on heating the solution to boiling a dark brown precipitate was formed (difference from a vanadate, which is not precipitated by hydrogen sulphide either in the cold or when heated), the filtrate from it being quite colourless (difference from a molybdate, which gives a blue coloured filtrate).

(2) On reducing the solution with zinc and hydrochloric acid no colour appeared for some time, but on standing a blue colour changing to brown was produced ; at the same time a substance having a metallic lustre adhered to the zinc. A solution of pure ammonium molybdate of the same concentration similarly treated at once turned blue, ultimately changing to brown, and the brown solution on long standing deposited a dirty brown amorphous substance.

(3) Barium chloride, lead acetate, silver nitrate, and mercurous nitrate all gave precipitates, soluble in nitric acid and similar in appearance to the respective salts of molybdic acid, except the mercurous salt. The barium salt forms a white crystalline powder, slightly soluble in water ; the lead salt is a white amorphous powder, insoluble in water ; the silver salt forms a pale yellow precipitate, slightly soluble in water ; and the mercurous salt consists of golden yellow crystals, insoluble in water. Mercurous molybdate is obtained, under like conditions, as a white or pale yellow amorphous substance. (Struve has observed that the milky precipitate of mercurous molybdate changes into golden yellow needles through prolonged washing or on standing.—*Journ. Prakt. Chem.*, lxi., 467, quoted in Dammer's "Handbook der Anorg. Chem., III., 628).

(4) After strongly acidifying the solution with nitric acid sodium phosphate gave a compound apparently similar to ammonium phosphomolybdate.

(5) In presence of potassium thiocyanate, zinc and hydrochloric acid gave a blood-red coloration.

Besides the above described reactions, which were observed with an aqueous solution of the salt, the following must also be mentioned.

(6) The residue obtained on heating the ammonium salt and consisting of the oxide, either alone or mixed with molybdenum trioxide, did not possess the characteristic yellowish red colour of vanadium pentoxide, but was similar in appearance to molybdenum trioxide and slowly volatilised at a red heat, becoming crystalline at the same time. It produced a deep blue coloration on heating with concentrated sulphuric acid.

(7) The oxide, when heated in a current of hydrogen gas, was reduced into the metal, which was non-fusible at a bright-red heat, and the metal could be easily brought back into the oxide by heating it in the air. The equivalent weight of the metal was determined both by reducing the oxide into the metal and by converting the metal back

into the oxide; the values obtained were 16·4 and 16·6, not very far from the equivalent weight of molybdenum.

Thinking that the ammonium salt above obtained might be a mixture of two salts, one containing molybdenum and the other the unknown element, an attempt was made to separate them, if possible, by fractional crystallisation. For this purpose the salt was again prepared according to the method already described, collecting, however, one crop of crystals shortly after the addition of ammonium chloride and without using alcohol, and another crop after adding alcohol to the mother-liquor and allowing it to stand for two days. From these two fractions, which looked exactly alike, water of crystallisation, ammonium oxide, and the residual oxide or oxides were determined with practically the same results.

The attempt which was next made to prepare the sodium salt resulted in showing the compound nature of the ammonium salt. The oxide obtained from the ammonium salt was fused with an equivalent quantity of sodium carbonate, calculated from the equivalent weight already determined on the supposition that the sodium salt would have the composition Na_2XO_4; the fused mass was dissolved in water and the solution evaporated, when hygroscopic crystals were obtained. Under a microscope these were found to consist partly of thin plates and partly of small needles, but, owing to their hygroscopic nature, their mechanical separation was found to be impossible.

Isolation of the element was at last effected through the electrolysis of the ammonium salt. On electrolysing an aqueous solution of the ammonium salt at a temperature of 60° to 70°, using a current density of 1 ampere per 100 sq. cm. and a potential difference of 4 volts, a black substance deposited on the cathode. It was found to contain molybdenum, doubtless as a hydrated sesquioxide, and on heating it with 2N-hydrochloric acid the molybdenum compound gradually dissolved away to form a dark brown solution. Treatment with hydrochloric acid was repeated until the fresh acid was no longer coloured brown. A portion of the residue (hydrated lower oxide of the new element ?), which was graphitic in appearance was boiled with nitric acid, the whole evaporated to dryness and extracted with ammonia. The ammoniacal solution was also evaporated to dryness and the residue dissolved in water. This aqueous solution gave all the reactions previously described, with the exception of the colour reactions mentioned in (2), (5), and (6), but these reactions are exactly those which are characteristic of molybdenum. It is true that a faint coloration was produced with zinc and hydrochloric acid in presence of potassium thiocyanate, but so faint that, considering the delicacy of this colour reaction, we may reasonably believe that almost every trace of molybdenum has been removed, and that the new element, or rather its oxide or the hydrated oxide, has been obtained in an almost pure state.

Properties of the Element.—The supposed new element, obtained by heating the above described graphitic substance in a current of hydrogen gas, has a metallic lustre and looks somewhat like magnesium powder. It burns brilliantly when heated in the air, producing the oxide, a part of which volatilises at the same time. The oxide, when heated in hydrogen gas, reproduces the metal in the form, however, of a grey powder resembling molybdenum, produced under like conditions. It is non-fusible at a red heat. On prolonged boiling it dissolves in concentrated hydrochloric acid, producing a faint greenish yellow solution. In this latter respect it is distinguished from molybdenum, which is insoluble in the acid. The hydrochloric acid solution on evaporation to dryness at 100° gives a white residue of the chloride, soluble in water. This residue, on ignition, becomes difficultly soluble, doubtless due to its conversion into the oxide or the oxychloride.

The equivalent weight was again determined, using the purified metal. (1) 0·1464 grm. of the metal gave 0·2149 grm. of the oxide, from which the number 17·1 was calculated. (2) 0·2149 grm. of the oxide gave 0·1451 grm. of the metal, which led to the value 16·7. Of these two results, the lower value, 16·7, which was obtained from the reduction of the oxide, must be regarded as more exact, there being an unavoidable slight loss by volatilisation of the oxide produced, when the metal is heated in the air, however carefully the heating be conducted. The reduction of the oxide can, on the contrary, be carried on with perfect safety, a lower oxide which is non-volatile at full red heat being, apparently, formed during the first stage of the reduction, so that if the heating is regulated at the beginning there is no danger of any loss being incurred.

The atomic weight of the element becomes 100, if calculated from the equivalent weight on the supposition that it is hexavalent like molybdenum. But the number 100 is that already given to nipponium as its atomic weight (*loc. cit.*), and nipponium differs from the element now under consideration in its oxide not being reducible by hydrogen and in other respects. It is possible that nipponium, instead of being divalent, as was supposed, may prove to be a trivalent element, with an atomic weight of 150, but discussion on this point, as well as on the atomic weight of the new element, will be reserved until the specific heat determinations of the two elements now being undertaken by Prof. Ikeda, of the Science College, and my analyses of their different compounds, shall have been completed.

It may be here stated, by way of parenthesis, that a new rich source of nipponium has been discovered. On carefully examining samples of thorianite there have been found here and there small, translucent, prismatic crystals, varying in colour from yellow to red. They are very hard and can scratch glass quite easily. The specific gravity of the mineral is 4·5, being about one-half of that of thorianite. This mineral was once thought to be zircon, but on further examination it has been found that it is a double silicate of zirconium and nipponium, and about 50 grms. of it have been picked out from 5 kilogrms. of thorianite. It is hoped that several grms. of nipponium may now be obtained from this source, about a grm. of the metal having been already prepared by the electrolysis of the double fluoride of nipponium and potassium.

Examination of the Sulphide Residue from Thorianite.

While in University College, London, Sir William Ramsay gave me for investigation 1¼ kilogrms. of crude hydrogen sulphide precipitate obtained from 5 cwt. of thorianite. This was ignited in the air, fused with hydrogen potassium sulphate, extracted with water, and re-precipitated by hydrogen sulphide. The filtrate from the precipitated sulphides was treated as in the ordinary course of analysis, and the solution, after the iron-group precipitate had been removed, had a deep reddish brown colour. It did not, however, produce any precipitate with ammonium sulphide, but the colour was suspected to be due to the presence of an unknown element. The sulphur, separated out from the solution on the addition of hydrochloric acid after ammonium sulphide had been added, was found to possess a deep brown colour and to leave a brown residue on ignition. About 100 grms. of this sulphur have been brought with me to Japan, and recently examined with the following results.

On ignition the sulphur gave about 8 per cent of the residue. This was repeatedly extracted with hot concentrated nitric acid, and it was found that the first portions of the extract had a bluish green and the later portions a reddish brown colour. Treatment with nitric acid was continued until no more colour was imparted to it, but a considerable fraction of the brown residue was still left undissolved.

The colour of the first portions of the extract was somewhat like that of a cupric salt, but that it was not due to the presence of this element was shown by the fact that the dark brown—almost black—precipitate produced by sodium sulphide, after nitric acid had been evaporated off, was completely dissolved by an excess of the reagent.

The sulphide, re-precipitated from the solution on the addition of hydrochloric acid, was moistened with concentrated nitric acid and ignited, and then fused with potassium nitrate. The brown residue left on extracting the fused mass with water was found to consist of an unknown oxide mixed with ferric oxide, it being this unknown oxide which dissolves in nitric acid with a bluish green colour. The aqueous extract did not contain any molybdenum, but it did contain the same new element as occurs in molybdenite, forming the subject of the present paper, as was shown by the formation of a brown sulphide, soluble in ammonium carbonate to form a deep brown solution, the precipitation of the salts of barium, lead, and silver, and more especially of the characteristic mercurous salt, as well as by the volatility and the crystalline nature of the oxide obtained from it.

The later portions of the nitric acid extract, which had a reddish brown colour, were evaporated to dryness, when a deliquescent, gummy mass of a brown colour was left behind. On ignition and subsequent fusion with potassium nitrate it was found to be resolved into the same three constituents as were obtained from the first portions of the nitric acid extract, namely, ferric oxide and the unknown oxide in the residue, after extracting the fused mass with water, this unknown oxide dissolving in nitric acid to form a bluish green solution, and the oxide of the new element in the aqueous extract. It must be here remarked that, although the nitric acid extracts, both green and brown, undeniably contain iron, as is shown by their subsequent treatment, they give no reactions with potassium ferrocyanide.

With regard to the unknown oxide, which dissolves in nitric acid with a bluish green colour, above referred to, no further observation has been made than that it is radio-active, and it is likely that the radio-activity of the sulphide residue from thorianite is due to the presence of this substance. It can be definitely stated that it does not contain any uranium. Since the brown coloured nitric acid extracts also give the same unknown oxide, dissolving in nitric acid with a bluish green colour after fusion with potassium nitrate, it is possible that the difference in colour may be due to the existence of two different oxides of one and the same unknown element.

Conclusions.

1. Both molybdenite and thorianite contain a new element, which is closely allied to molybdenum.

2. It forms a complex salt (?) with ammonium molybdate, precipitable, from its aqueous solution, in a crystalline form on saturation with ammonium chloride.

3. It appears to form two different oxides, the higher oxide being similar in its properties to molybdenum trioxide and acting as an acidic oxide. The salts of barium, lead, and silver formed from the higher oxide resemble the corresponding molybdates, but the mercurous salt consists of golden-yellow crystals.

4. The hydrated lower oxide (?) has a graphitic appearance, and is insoluble in hot dilute hydrochloric acid.

5. On heating in a current of hydrogen gas, either of the oxides produces the metal, which is non-fusible at a red heat. It burns brilliantly in the air, producing the higher oxide.

6. The equivalent weight of the metal is somewhat above that of molybdenum, being about 16·7.

7. In producing a brown sulphide and in other respects, above referred to, the new element resembles molybdenum, but is distinguished from the latter in producing no colour reactions, when a soluble salt of its higher oxide is reduced with zinc and hydrochloric acid, either in the presence or absence of potassium thiocyanate, and also by the fact that the metal dissolves in hot concentrated hydrochloric acid.

8. The "new tin-group element," recently described by Miss Evans, is probably identical with the element forming the subject of this paper.

9. A new rich source of nipponium has been discovered and, incidentally, described.

10. Besides nipponium and the new element now under consideration, thorianite appears to contain still another new element, the oxide of which is radio-active and dissolves in nitric acid to form a bluish green solution.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 252).

Confirmatory Experiments and References (continued).

Delicacy of the Confirmatory Test for Chromate.—To a solution containing 0·5 cc. Cr as K_2CrO_4 in 100 cc. was added a little ammonium acetate solution, 2 cc. acetic acid, and 1 grm. $BaCl_2$: a yellow precipitate separated at once. This was collected on a filter, and treated by P. 56: upon the addition of H_2O_2 and ether, a distinct blue colour resulted. The experiment was repeated with 0·2 mgrm. Cr: a slight but distinct precipitate and colour were obtained.

Reduction of Chromic Acid by Filter-paper.—10 mgrms. Cr as K_2CrO_4 were precipitated as lead chromate, and the mixture filtered. The precipitate, with the filter, was placed in a casserole; 10 cc. water and 5 cc. HNO_3 (1·20) were added, and the mixture was boiled. The solution turned green in about one minute, thus showing that reduction takes place readily.

P. 57, N. 3: *Ignition Test for Zinc with Cobalt Nitrate.*—0·1, 0·2, and 10 mgrms. Zn as $Zn(NO_3)_2$ were treated as described in the second paragraph of P. 57, 0·2 mgrm. Co as nitrate being used in each experiment: a distinct green colour resulted in the experiment with 0·1 mgrm. Zn, a deeper colour with 0·2 mgrm. and an intense colour with 10 mgrms. 1 mgrm. Zn and 2 mgrms. Co as nitrates were treated as described in the last paragraph of P. 57: the residue was green. The residue was then heated strongly: it turned black, showing that when an excess of cobalt is present the test is more delicate if the mixture is not heated strongly. The experiment was repeated with 2 mgrms. Cr and 1 mgrm. Zn: a green colour resulted which remained after the residue had been heated strongly.

A solution containing 5 mgrms. Zn and 0·5 mgrm. Co as nitrates was evaporated to dryness in a casserole and the mixture was ignited, first gently and then strongly: the residue was black owing to the presence of cobalt oxide. 0·3 mgrm. Zn and 0·2 mgrm. Co as nitrates were treated by the last paragraph of P. 57, except that 5 cc. of 10 per cent Na_2CO_3 solution were added in excess: only a very faint green colour resulted. The experiment was repeated except that only 3 drops Na_2CO_3 solution were added in excess; the green colour was mixed with black. The experiment was repeated, with an excess of 1 cc. and also with an excess of 0·5 cc. Na_2CO_3 solution: a good green colour was obtained. These experiments show that some Na_2CO_3 must be added, but only a moderate quantity.

A solution containing 0·3 mgrm. Zn and 0·2 mgrm. Co as chlorides and an excess HCl was treated by P. 57 (last par.), no HNO_3 being added: the residue had no distinct colour, showing that HCl cannot be substituted for HNO_3.

*P. 58*a*, N. 3: *Composition of the Zinc and Beryllium Precipitates produced by Sodium Hydrogen Carbonate.*—500 mgrms. Zn as nitrate were treated by *P. 58*a*, using a total volume of 100 cc. The precipitate was carefully washed with water, and tested for carbonate in the usual way by treating with acid in a flask and passing the gas

* From the *Journal of the American Chemical Society*, XXX., No. 4.

evolved through $Ba(OH)_2$ solution. A large precipitate of $BaCO_3$ was obtained which corresponded to at least 200 to 300 mgrms. Zn. The experiment was repeated with 200 mgrms. Be: a large amount of carbonate was also found in the beryllium precipitate, the precipitate of $BaCO_3$ being about twice as large as in the experiment with zinc. Parsons states (*Journ. Am. Chem. Soc.*, 1906, xxviii., 557) that the precipitate obtained in a dilute ammonium carbonate solution is a basic carbonate of beryllium.

Precipitation of Aluminium in Hot Sodium Hydrogen Carbonate Solutions in a Closed Bottle.—Solutions containing 2 mgrms. Al as nitrate in 100 cc. were placed in pressure bottles, and 2, 5, and 10 mgrms. $NaHCO_3$ added: each of the three solutions quickly became turbid in the cold. The bottles were heated at about 95° in a water-bath for half an hour: a flocculent precipitate formed in each case in less than five minutes, and settled in less than twenty minutes. The bottles were cooled and the precipitates filtered off; the filtrates were evaporated with excess of HNO_3 and tested for aluminium with NH_4OH: little or no precipitate separated, showing that the precipitation of the aluminium had been practically complete in each case. The experiments were repeated with 1, $\frac{1}{2}$, $\frac{1}{10}$, and $\frac{1}{20}$ grms. $NaHCO_3$; the results were the same except that the precipitation was not complete with $\frac{1}{20}$ grm. $NaHCO_3$. The experiment was repeated with $\frac{1}{14}$ grm. $NaHCO_3$; no precipitate was obtained. These experiments were repeated, except that the solution was thoroughly saturated with CO_2 gas before adding the $NaHCO_3$; the results were nearly the same.

A solution containing 500 mgrms. Al as nitrate in 100 cc. was placed in a bottle and 4 grms. $NaHCO_3$ added; there was a violent evolution of CO_3 gas, a small precipitate formed, and the solution still reacted acid to litmus. One grm. more $NaHCO_3$ was added; a very large precipitate separated, very little gas came off, and the solution after shaking did not turn blue litmus red. The mixture was heated in the closed bottle at 95° in a water-bath for half an hour, cooled, and filtered; the filtrate was found to contain no aluminium. Therefore 500 mgrms. Al are completely precipitated when about 5 grms. $NaHCO_3$ are added in 100 cc. of solution.

Precipitation of Zinc Carbonate in Hot Sodium Hydrogen Carbonate Solutions.—Four solutions, each containing 2 mgrms. Zn as nitrate dissolved in 100 cc., were poured into strong 200 cc. bottles; to these were added 1, 2, 3, and 5 grms. respectively of solid $NaHCO_3$: the bottles were corked and shaken until the $NaHCO_3$ dissolved; they were then heated in a water-bath at 90° to 95° for half an hour; all the solutions became slightly turbid in the cold and after heating for five minutes precipitates could be seen in suspension. After the half hour's heating the bottles were allowed to cool to about 40°, and the solutions containing 1 and 5 grms. $NaHCO_3$ were filtered and tested for zinc by evaporating with HNO_3, adding NH_4OH and acetic acid, and passing in H_2S; only a trace of zinc was found in each filtrate, thus showing that the precipitation was practically complete in both 1 per cent and 5 per cent $NaHCO_3$ solutions. The experiments with 1 and 2 grms. $NaHCO_3$ were repeated except that the solutions were thoroughly saturated with CO_2 gas at room temperature before the $NaHCO_3$ portions were added: the results were the same.

The experiment was repeated with a solution containing 500 mgrms. Zn as nitrate in 100 cc., 2 grms. $NaHCO_3$ being added; no precipitate of ZnS separated. The experiment was repeated with 500 mgrms. Zn and 1·5 grms. $NaHCO_3$; about 5 mgrms. Zn were found in the filtrate, showing that 1·5 grms. $NaHCO_3$ is not quite sufficient to precipitate 500 mgrms. Zn completely.

Precipitation of Beryllium in Hot Sodium Hydrogen Carbonate Solutions in a Closed Bottle.—A solution containing 1 mgrm. Be as nitrate in 100 cc. was placed in a 200 cc. bottle, 1 grm. $NaHCO_3$ was added, and the mixture was digested in the tightly stoppered bottle at about 95° in a water-bath for half an hour; the solution became turbid in less than five minutes and a precipitate separated in ten minutes; on standing in the cold for an hour the precipitate seemed to re-dissolve to a small extent. The experiment was repeated with a solution that was saturated with CO_2 gas before the $NaHCO_3$ was added; the result was the same. These experiments were repeated with 1 mgrm. Be and 2 grms. $NaHCO_3$; the mixture that had been saturated with CO_2 was distinctly turbid in fifteen minutes, while the other was only very slightly turbid after half an hour. The solutions cleared on standing for an hour in the cold. The last experiments were repeated with 2 mgrms. Be and 2 grms. $NaHCO_3$: distinct precipitates were obtained corresponding to about 1 mgrm. Be, but these re-dissolved to a considerable extent in the cold.

These experiments were repeated with solutions containing 3 to 10 grms. $NaHCO_3$ in 100 cc. of solution and varying quantities of beryllium; in the 3 per cent $NaHCO_3$ solution 3 mgrms. Be gave no precipitate, but a larger amount did so; in the 5 per cent solution, about 15 mgrms. Be just remained in solution in 100 cc.; and in the 10 per cent solution, about 75 mgrms. Be gave a small precipitate in 50 cc., while 50 mgrms. Be gave only a negligible precipitate.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 5th, 1908.

Sir WILLIAM RAMSAY, K.C.B., F.R.S., President, in the Chair.

MR. G. J. FOWLER was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. Thomas James Baker, D.Sc., Trebarwith, Chester Road, Erdington, Birmingham; Charles Everitt, M.A., 67, Boston Avenue, Southend-on-Sea; Henry Hamilton Green, 36, Princes Square, Regent Park, Glasgow; Edward Hinks, B.Sc., 23, Tavistock Road, Croydon; Joseph Kenyon, B.Sc., 6, Cheltenham Street, Blackburn; Gregor MacGregor, M.A., B.Sc., Giffen Park, Dysart, Fife; William Herbert Miles, 3, Liversidge Road, Higher Tranmere, Birkenhead; Charles James Millar, care of Messrs. Turner, Morrison, and Co., Calcutta; George Edward Pearson, care of Messrs. Burroughs, Wellcome, and Co., Snow Hill Buildings, E.C.; Frank Moreton Thomas, 290, Rotton Park Road, Edgbaston, Birmingham; Sidney Gilbert Walton, "Kensington," Northwood Road, Northwood, Lane Cove River, N.S.W.; William Crd Wootton, B.Sc., 159, Maidstone Road, Rochester; Frederick Palliser Worley, M.A., M.Sc., Mansfield House, Clifton Gardens, Maida Hill, W.

Of the following papers, those marked * were read:—

*221. "*The Direct Union of Carbon and Hydrogen. Synthesis of Methane,*" By WILLIAM ARTHUR BONE and HUBERT FRANK COWARD.

The recent work of Berthelot, Pring and Hutton, and Mayer and Altmayer on this subject was critically reviewed. The results of new experiments were described which demonstrate the formation of large quantities of methane when less than 0·1 grm. of sugar-charcoal (highly purified by prolonged ignition in dry chlorine, first of all at 700° and then at 1100°) is heated in a stream of pure dry hydrogen to between 1100° and 1200° in an apparatus similar to that used by Bone and Jerdan in 1897.

In two separate experiments, the yields of methane (namely, 137·0 cc. and 125·5 cc. at 0° and 760 mm. from 0·0995 and 0·092 grm. of carbon respectively) were as much as 73 per cent of those theoretically obtainable, the

remainder of the methane being lost by unavoidable oxidation or decomposition in contact with the intensely hot walls of the porcelain tube in which the carbon was heated. In one group of four experiments, the formation of altogether 422 cc. of methane from 0·306 grm. of carbon was demonstrated. The authors therefore consider that the synthesis of methane from its elements, as originally carried out by Bone and Jerdan in 1897 and 1901, is fully confirmed.

Incidentally, the results of analyses of the sample of highly purified carbon originally used by Bone and Jerdan were given, showing that the hydrogen in it did not exceed 0·06 per cent, of which at least part would be occluded.

Discussion.

Dr. Lowry, as evidence of the unreliability of thermodynamic calculations of high temperature equilibria, referred to the case of nickel oxide, where the pressure of oxygen at 1127° had been calculated by Stahl (*Metallurgie*, 1907, iv., 682) as 0·000000033 mm., the values actually observed (Foote and Smith, *Journ. Am. Chem. Soc.*, 1908, xxx., 1349) being 5·5 mm. at 1100° and 13·0 mm. at 1200°. The value calculated for 227° was $1{\cdot}859 \times 10^{-45}$, or roughly $0{\cdot}5 \times 10^{-21}$ molecule per 22·4 litres, and in the case of zinc oxide, $1{\cdot}333 \times 10^{-67}$ or $0{\cdot}3 \times 10^{-43}$ molecule, figures which, in spite of the formal exactness, were of such ultra-electronic minuteness as to have no significance whatever.

Dr. Feilmann asked whether Prof. Bone could supply information as to the black substance formed by the methane on the walls of the porcelain tube in which the reaction took place, and suggested that it might contain carbon, due to decomposition of the methane formed; such decomposition might be promoted both by the walls of the tube being presumably rather hotter than the body of the gas, and also by the special action which was well known to be peculiar to solid surfaces in similar circumstances. It was difficult to believe that the coloration could be due to the formation of silicon in the porcelain, and it presumably contained no lead.

Prof. Bone, in reply, wished it to be understood that he did not attack Nernst's thermodynamical arguments as being, from the abstract point of view, unsound—this not being the point raised in the paper—but merely contended that for calculating high temperature equilibria they were at present useless, owing to defective knowledge of the measure of the thermal factors involved. There was nowadays a tendency, in certain quarters, to over-estimate the value of mathematical deductions in relation to chemical phenomena, and it was necessary to protest against the practice of setting aside the results of carefully conducted experiments on merely mathematical grounds.

In answer to Dr. Feilmann, he was afraid he could give no definite information as to what particular constituent of the porcelain or the glaze of the inner tube had been reduced by the methane; presumably it might be the silica, and certainly the appearance of the tube suggested this. With regard to the mechanism of the process, it might be either a direct reduction by methane itself, or a secondary effect due to carbon liberated by a primary decomposition of methane in contact with the hot surface.

*222. "*The Relation between Absorption Spectra and Chemical Constitution.* Part XI. *Some Aromatic Hydrocarbons.*" By Edward Charles Cyril Baly and William Bradshaw Tuck.

The authors have examined the absorption spectra of the following hydrocarbons:—Naphthalene, acenaphthene, acenaphthylene, fluorene, chrysene, anthracene, phenanthrene, diphenyl, and dibenzyl. Certain minor differences from Hartley's observations of naphthalene, anthracene, and phenanthrene (*Trans.*, 1881, xxxix., 153; 1885, xlvii., 685) have been noted. In some cases the absorption of the hydrocarbons shows considerable similarity among themselves, from which it has been found possible to gain some insight into the dynamic condition of these substances.

*223. "*Organic Derivatives of Silicon.* Part VII. *The Synthesis of* dl-*Sulphobenzylethylisobutylsilicyl Oxide.*" By Bernard Dunstan Wilkinson Luff and Frederic Stanley Kipping.

The only optically active silicon compounds which have hitherto been described, namely, the *d*- and *l*-forms of sulphobenzylethylpropylsilicyl oxide, $SO_3H{\cdot}C_6H_4{\cdot}CH_2{\cdot}SiEtPr{\cdot}O{\cdot}SiEtPr{\cdot}CH_2{\cdot}C_6H_4{\cdot}SO_3H$, have very low specific rotations, and in many cases when they are combined with a given optically active base their enantiomorphous relationship is not very clearly indicated.

Although there can be no doubt that the molecules of these acids are asymmetric, it seemed desirable to prepare, if possible, some other compound of the same type. With this object, the authors have synthesised *benzylethylisobutylsilicyl chloride*, $SiEt(C_7H_7)(C_4H_9)Cl$, b. p. 198 — 202° (100 mm.); *benzylethylisobutylsilicol*, $SiEt(C_7H_7)(C_4H_9){\cdot}OH$, b. p. 162—164° (25 mm.), and *benzylethylisobutylsilicyl oxide*, $[SiEt(C_7H_7)(C_4H_9)]_2O$, b. p. 250—252° (20 mm.). Each of these three substances was then sulphonated with the aid of chlorosulphonic acid, and also in the case of the silicol and the oxide with concentrated sulphuric acid. The product in all cases was a mixture from which the same *dl*-acid, namely, dl-*sulphobenzylethylisobutylsilicyl oxide*, was isolated in the form of its l-*menthylamine* salt; this compound, $[SiEt(C_4H_9){\cdot}CH_2{\cdot}C_6H_4{\cdot}SO_3H]_2O,2C_{10}H_{21}N,4H_2O$, melts at 240—245°, and its specific rotation is $[\alpha]_D$ −14·7° in methyl-alcoholic solution; it is not resolved by fractional crystallisation.

From the *l*-menthylamine derivative, the *sodium* salt of the pure *dl*-acid was obtained, and from the latter the d-*bornylamine*, *cinchonidine*, and *cinchonidine hydrogen* salts were prepared.

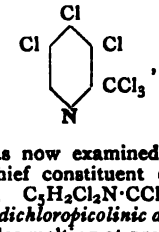
Experiments on the resolution of this *dl*-acid will be described at an early date.

*224. "*The Relation between Viscosity and Chemical Constitution.* Part III. *The Enol-ketonic Tautomerism.*" By Albert Ernest Dunstan and James Arthur Stubbs.

The authors have investigated the viscosity of ethyl acetoacetate, ethyl ethylacetoacetate, and ethyl diethylacetoacetate, alone and in various solvents. They have also studied the effects of piperidine and sodium ethoxide on ethyl acetoacetate. In confirmation of the work of Brühl, Perkin, Drude, Schaum, Knorr, and Lowry on these and similar compounds, they conclude that ethyl acetoacetate is mainly existent in the ketonic condition.

225. "*The Chlorination of Methyl Derivatives of Pyridine.* Part II. 2-*Methylpyridine.*" By William James Sell.

When α-picoline hydrochloride saturated with hydrogen chloride is chlorinated at 105—110° until no further action is apparent, an oily mixture of chlorinated derivatives is obtained, which, on the removal of the excess of chlorine and hydrogen chloride by a current of dry air, partly solidifies. The solid compound thus obtained was shown (Part I., *Trans.*, 1905, lxxxvii., 799) to have the constitutional formula:—



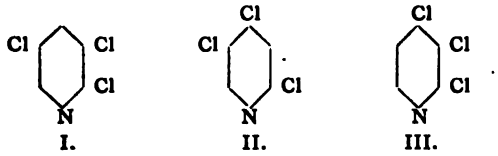
and the author has now examined the liquid part of the products. The chief constituent of this oily liquid is *pentachloropicoline*, $C_5H_2Cl_2N{\cdot}CCl_3$. This compound readily yields 3 : 5-*dichloropicolinic acid*, which crystallises from water in needles melting at 153—154°. The *methyl* ester and the *amide* have been prepared. On heating, the acid is converted into 3 : 5-*dichloropyridine*, m. p. 66—67°.

226. "*The Chlorine Derivatives of Pyridine.* Part IX. *Preparation and Orientation of* 3 : 5-*Dichloropyridine.*" By William James Sell.

The well-marked basic characters of this substance, together with its great stability, suggested that the chlorine atoms in this compound occupy the 3 : 5-position. This conclusion is strengthened by the analogy in general properties and stability under similar conditions to the dibromopyridine, m. p. 110—111°, in which the bromine atoms are known to occupy the positions 3 : 5. The orientation was carried out by converting specimens of the 3 : 5-dibromopyridine and the dichloropyridine in question into diethoxypyridine, and showing that the diethoxypyridines from the two sources yield identical compounds with bromine and with chlorine.

227. "*The Chlorine Derivatives of Pyridine.* Part X. *Orientation of* 2 : 3 : 5-*Trichloropyridine.*" By WILLIAM JAMES SELL.

Excluding the trichloropyridines of known constitution, and having regard to the feeble, but distinct, basic properties of the compound in question, there seemed every probability that its constitution should be represented by one of the following formulæ :—



I. II. III.

of which I. was regarded as sufficiently probable to warrant the attempt to determine its constitution on the following lines :—

Such a compound ought to be attacked by ammonia, giving 3 : 5-dichloro-2-aminopyridine. This was carried out, and a substance melting at 84—85° was isolated. Further, 3 : 5-dichloropicolinamide (*Proc. Chem. Soc.*, xxiv., 225) was converted by the Hofmann reaction into 3 : 5-dichloro-2-aminopyridine.

The compound thus prepared melted at 84—85°, and the melting-point was unaffected by admixture with the specimen obtained by the action of ammonia on the trichloropyridine in question. Moreover, Königs and Geigy have described (*Ber.*, 1884, xvii., 589, 1833) a dichloroethoxypyridine obtained from this trichloropyridine by the action of sodium ethoxide. This substance was converted into the dichlorohydroxypyridine, m. p. 178°, by heating with hydrochloric acid.

Finally, 3 : 5-dichloro-2-aminopyridine was converted by a modification of the Bouveault reaction into the same dichlorohydroxypyridine, m. p. 178—179°, as was obtained from the trichloropyridine in question.

228. "*The Triazo-group.* Part V. *Resolution of α-Triazopropionic Acid.*" By MARTIN ONSLOW FORSTER and HANS EDUARD FIERZ.

l-α-*Triazopropionic acid*, $CH_3 \cdot CHN_3 \cdot CO_2H$, has the specific gravity 1·2293 compared with water at 21°, and $[\alpha]_D$ −2·27°; a 16 per cent solution in ether has $[\alpha]_D$ −28·9°, and a 21 per cent solution in water, $[\alpha]_D$ +4·8°. The *brucine* salt, $C_{26}H_{31}O_6N_5$, has not a definite melting-point, but evolves gas at about 150°; the aqueous solution displays mutarotation, the permanent value being $[\alpha]_D$ −26·4° in a concentration of 4 per cent. The *ethyl* ester, $C_5H_9O_2N_3$, boils at 49° under 5 mm. pressure, has specific gravity 1·063 compared with water at 22°, and $[\alpha]_D$ −18·2°; the *amide*, $C_3H_6ON_4$, melts at 85°, and is dextrorotatory, having $[\alpha]_D$ 49·1° in water. When l-α-triazopropionic acid is reduced with aluminium amalgam, d-alanine is formed.

229. "*The Triazo-group.* Part VI. *Triazoethyl Alcohol and Triazoacetaldehyde.*" By MARTIN ONSLOW FORSTER and HANS EDUARD FIERZ.

Triazoethyl alcohol (2-*triazoethanol*-1), $N_3 \cdot CH_2 \cdot CH_2 \cdot OH$, prepared from ethylene chlorohydrin and sodium azide, is a colourless, sweet-tasting, odourless liquid, having specific gravity 1·149 compared with water at 24°, and boiling at 60° under 8 mm. pressure; treatment with stannous chloride eliminates nitrogen, and produces aminoethyl alcohol. *Triazoethyl acetate*, $CH_3 \cdot CO_2 \cdot C_2H_4N_3$, has specific gravity 1·126 compared with water at 24°, and boils at 74° under 20 mm. pressure; the odour is much more pronounced than that of ethyl triazoacetate, resembling that of ethyl acetate in character and intensity.

Triazoethyl p-*nitrobenzoate*, $NO_2 \cdot C_6H_4 \cdot CO_2 \cdot C_2H_4N_3$, melts at 82°, and, on reduction with tin and hydrochloric acid, yields p-*aminobenzoyloxyethylamine*, $NH_2 \cdot C_6H_4 \cdot CO_2 \cdot C_2H_4 \cdot NH_2$, which melts at 78° when crystallised with $1H_2O$, the anhydrous base melting at 122·5°; the *picrate* and *dibenzoyl* derivative melt at 172—175° and 212° respectively.

Triazoacetaldehyde, $N_3 \cdot CH_2 \cdot CH{:}O$, obtained from chloroacetaldehyde hydrate and sodium azide, is an oil, which has not been purified on account of the readiness with which it undergoes change on heating, or when treated with various agents; its behaviour towards alkalis resembles that of the triazo-ketones.

230. "*Contribution to the Chemistry of the Cholesterol Group.* Part II. *Some Oxidation Products of Sitosterol.*" By ROBERT HOWSON PICKARD and JOSEPH YATES.

The behaviour on oxidation of the vegetable cholesterol, sitosterol, has been studied. This alcohol, which is obtained from the fat contained in wheat, yields oxidation products which are analogous to those given by cholesterol itself.

Using a nomenclature similar to that of Windaus (*Arch. Pharm.*, 1908, [2], ccxlvi., 117) for cholesteryl derivatives, the following are the products obtained :—By the action of hydrogen peroxide on sitosterol, *dehydrositostanetriol*, $C_{27}H_{46}O_3$, m. p. 252°, and its *diacetate*, m. p. 138—139°; by the action of chromium trioxide on the triol, *dehydrositostanedionol*, $C_{27}H_{42}O_3$, m. p. 256°; by the action of dehydrating agents on the dionol *dehydrositostenedione*, $C_{27}H_{40}O_2$, m. p. 166°, and its *monophenylhydrazone*, m. p. 247°; by the action of zinc-dust on the unsaturated dione, *dehydrositostanedione*, $C_{27}H_{42}O_2$, m. p. 196°, and its *dioxime*, m. p. 187°; by the oxidation of the saturated dione with chromium trioxide, a *ketodicarboxylic acid*, $C_{27}H_{42}O_5$, m. p. 236°, and its *dimethyl* ester, m. p. 107°, and also the *oxime* of the ester, m. p. about 90°.

231. "*Experiments on the Synthesis of the Terepenes.* Part I. (cont.). *Resolution of* dl-1-*Methyl*-Δ^1-cyclo*hexene*-4-*carboxylic Acid and Synthesis of the Optically Active Modifications of Terpineol.*" By KENNETH FISHER and WILLIAM HENRY PERKIN, jun.

The authors have synthesised *d*- and *l*-terpineol from the esters of the *d*- and *l*-modifications of 1-methyl-Δ^1-*cyclo*-hexene-4-carboxylic acid, $CMe\begin{matrix}=CH—CH_2\\ \diagdown CH_2 \cdot CH_2\end{matrix}\!\!>CH \cdot CO_2H$, and have attempted to convert them into the optically active modifications of limonene.

232. "*Experiments on the Synthesis of the Terpenes.* Part XIII. *Synthesis of* iso*Carvestrene* ($\Delta^{6:8(9)}$-m*ethadiene*) *and its Derivatives.*" By KENNETH FISHER and WILLIAM HENRY PERKIN, jun.

The authors have synthesised 1-*methyl*-Δ^6-cyclo*hexene*-3-*carboxylic acid*, and have converted it into *dihydro*iso*carvestrenol* (I.) and iso*carvestrene* (II.) :—

$$CH\begin{matrix}=CMe \cdot CH_2\\ \diagdown CH_2 \cdot CH_2\end{matrix}\!\!>CH \cdot CMe_2 \cdot OH$$

I.

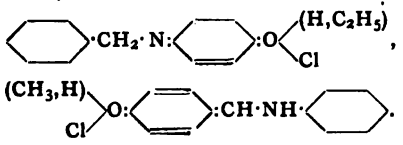
$$CH\begin{matrix}=CMe \cdot CH_2\\ \diagdown CH_2—CH_2\end{matrix}\!\!>CH \cdot CMe{:}CH_2.$$

II.

233. "*Colour and Constitution of Azomethine Compounds.*" (Part II.). By FRANK GEORGE POPE and ROBERT FLEMING.

The simplest hydroxyazomethine compounds and their ethers are colourless substances, but their hydrochlorides, which are formed by passing hydrogen chloride into benzene solutions of the azomethines, show absorption in

the blue and violet region of the visible spectrum. Consequently, it seems necessary to conclude that the salts formed by the action of mineral acids on the azomethines possess a different structure from the azomethines themselves, and it appears most rational to regard them as oxonium salts, thus:—

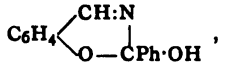


234. "*Aromatic Arsonic Acids.*" By MARMADUKE BARROWCLIFF, FRANK LEE PYMAN, and FREDERIC GEORGE PERCY REMFRY.

Sodium *bis*-2-aminotolyl-5-arsinate described by Pyman and Reynolds (*Trans.*, 1908, xciii., 1180) is of little use in trypanosomiasis. The authors have prepared several derivatives of *p*-aminophenylarsonic acid and 2-aminotolyl-5-arsonic acid for possible use in trypanosomiasis; their trypanocidal properties were found to be very slight.

235. "*The Condensation of Salicylaldehyde and Benzamide.*" By ARTHUR WALSH TITHERLEY and MORRIS EDGAR MARPLES.

The condensation of salicylaldehyde and benzamide in the presence of sodium acetate produces two isomeric *salicylidenebenzamides*, $HO\cdot C_6H_4\cdot CH:N\cdot CO\cdot C_6H_5$, and not the cyclic "coumarazine,"—



described by Cebrian (*Ber.*, 1898, xxxi., 1592). The two compounds, which are similar in properties and phenolic in character, were separated by means of acetone.

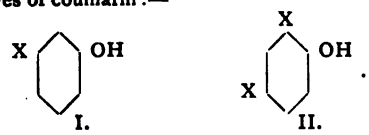
When condensation between salicylaldehyde and benzamide occurs in presence of hydrogen chloride, wandering of benzoyl occurs, and O-N-N-*tribenzoylsalicylidenediamine*, $BzO\cdot C_6H_4.CH(NHBz)_2$, m. p. 198°, is formed, which is hydrolysed by pyridine and alkali, forming ammonia, benzoic acid, salicylaldehyde, and dibenzamide. The production of the latter shows that a wandering of benzoyl takes place similar to that which occurs in the synthetic formation of the tribenzoyl derivative.

By cautious hydrolysis of the tribenzoyl derivative with cold aqueous alcoholic sodium hydroxide, one benzoyl group is eliminated with the production of N-N-*dibenzoylsalicylidenediamine*, $HO\cdot C_6H_4\cdot CH(NHBz)_2$, m. p. 192°.

236. "*The Coumarin Condensation.*" By ARTHUR CLAYTON.

A study has been made of the formation of coumarins from substituted phenols and malic acid or ethyl acetoacetate. The following conclusions are drawn:—

1. Phenols of the types shown in formulæ I. and II. (where X is an alkyl, hydroxyl, or dialkylamino group) condense readily with malic acid or ethyl acetoacetate to form derivatives of coumarin:—



2. Chlorine as a substituent has an effect similar to that of the above groups, but its influence is very much less marked.

3. The introduction of such substituents as NO_2, CO_2H, and $CO_2\cdot C_2H_5$ prevents condensation.

237. "*Experiments on the Synthesis of* 1-*Methylcyclohexylidene-4-acetic Acid.*" (Part II.). By VICTOR JOHN HARDING, WALTER NORMAN HAWORTH, and WILLIAM HENRY PERKIN, jun.

The authors bring forward evidence which, in their opinion, clearly proves that the acid of melting-point 41°, obtained by Marckwald and Meth (*Ber.*, 1906, xxxix., 1171) by the elimination of water from 4-hydroxy-1-methyl-*cyclo*hexyl-4-acetic acid,—

$$CHMe\begin{matrix}CH_2\cdot CH_2\\CH_2\cdot CH_2\end{matrix}>C(OH)\cdot CH_2\cdot CO_2H,$$

is 1-methyl-Δ^3-*cyclo*hexene-4-acetic acid,—

$$CHMe\begin{matrix}CH_2—CH\\CH_2\cdot CH_2\end{matrix}\geqslant C\cdot CH_2\cdot CO_2H,$$

and not, as the above investigators claim to have established, 1-methyl*cyclo*hexylidene-4-acetic acid,—

$$CHMe\begin{matrix}CH_2\cdot CH_2\\CH_2\cdot CH_2\end{matrix}>C:CH\cdot CO_2H.$$

PHYSICAL SOCIETY.

Ordinary Meeting, November 13th, 1908.

Dr. C. CHREE, F.R.S., President, in the Chair.

THE meeting was held, by kind invitation of Prof. F. T. Trouton, in the Physical Laboratory at University College.

Dr. FLEMING communicated a note on the "*Photo-electric Properties of Potassium-soaium Alloy*," illustrated by experiments.

The author said that, in connection with further researches on radio-telegraphic wave-detectors of the oscillation valve type, he had been led to examine instances of electronic emission other than those due to the incandescence of metals or carbon, to ascertain how far rectifying effects with high frequency currents could be obtained by them. It is well known that under the action of ordinary and ultra-violet light the electro-positive metals lose a negative charge of electricity, the effect being most pronounced in the case of rubidium, potassium, and the liquid alloy of potassium-sodium.

Dr. Fleming described a mode of making a tube in which potassium and sodium could be melted together and then decanted over into a chamber containing a platinum plate, so that a mass of the liquid potassium-sodium alloy having a perfectly clean surface, and the platinum plate fixed above it in an inclined position, could be constructed. By means of platinum wires sealed through the glass tube, a contact is made respectively with the platinum plate and the liquid alloy. When the surface of the alloy is illuminated by a powerful beam of light and the galvanometer connected to the platinum plate and alloy, it is found that a current is produced in this circuit merely by the action of the light, and by balancing this current against a variable electromotive force it was shown that an electromotive force varying from 0·4 to 0·8 of a volt is produced in the photo-electric cell due to the action of the light.

It was then shown that this photo-electric effect was greatly dependent an the colour of the light, and especially upon light absorbed by the alloy. The most effective rays in the case of potassium-sodium alloy having in it some excess of potassium, are the violet rays which can pass through cobalt glass, but even a very thin sheet of yellow glass cuts off the effect entirely. Elster and Geitel's experiment was then shown illustrating the effect of the plane of polarisation of the incident light. A powerful beam of light polarised by a Nicol prism was thrown upon the alloy's surface, and the self-produced photoelectromotive force was shown to be a maximum when the plane of polarisation of the beam was perpendicular to the plane of incidence of the light, thus showing that a maximum effect of the light is produced when the electric vector in the ray is perpendicular to the surface of the alloy, but a minimum when it is at right angles.

It was then shown that the photo-electric effect is greatly diminished by making even a feeble magnetic field parallel to the surface of the alloy. By the use of two such cells placed in series, Dr. Fleming showed that the photo-

electromotive force of each were additive, and indicated the possibility of making a battery of high voltage by the action of light upon a rubidium surface. Such a cell has more or less unilateral conductivity, and can therefore be used to rectify high frequency currents, but the effect is inferior to that produced by the author's oscillation valves, particularly by his latest type of valve.

Some questions were then discussed tending to show that the source of the photo-electromotive force cannot be identical with the volta-contact electromotive force in the circuit, but is due to the absorption of light by the sensitive surface. In all cases of such photo-electric action, the rays which are effective in producing it are those given out by the substance if heated or otherwise made radiant. Thus, zinc is photo-electric under the action of ultra-violet light, and ultra-violet light is largely radiated when an electric spark is taken between the zinc balls, or an electric arc between zinc poles. The photo-electric effects in the case of potassium are probably due to the absorption of the violet ray emitted by potassium when heated.

Some points of interest in connection with radio-telegraphy were then discussed. It has been shown that perfectly dust-free air is not ionised by ultra-violet light. If therefore the absorption of radio-telegraphic electric waves, which is found to exist when they pass through long distances of sunlit air, is due to the presence of free ions in the air, this may arise from the photo-electric action of the light upon dust particles. This suggests the question whether these particles may not be the same which create the blue colour of the sky, and if these particles absorb ultra-violet light, and thereby create ionisation, that would account for the relatively small percentage of ultra-violet light found in sunlight at the earth's surface, and also suggests the need for further observations on the principle of ions present in the atmosphere at various heights.

Prof. S. P. THOMPSON asked the author whether sodium was more sensitive to yellow light than to light of other wave-lengths,

Dr. R. S. WILLOWS remarked that the E.M.F. was in the direction required by theory: the alloy emits negative ions, and hence the direction of flow of negative electricity in the cell is from the alloy to the platinum, or the alloy is the positive pole. The action was similar in results to those produced by solution pressure when metals were immersed in acids except that the ions produced by light were of the other sign. Prof. Fleming's idea that metals respond to those rays only which they absorb was interesting in view of the fact that the weight of evidence was in favour of the emission of ions being due to an explosion in the atom arising from resonance, but Ladenburg's experiments did not support Dr. Fleming's theory. He measured the intensity of the light with a thermo-couple, and found for the same intensity that the shorter the wave-length the more efficient was the light in producing emission of ions.

Dr. FLEMING, in reply to Prof. Thompson, said sodium was most sensitive to yellow light.

A paper entitled "*Electric Splashes on Photographic Plates*" was read by Mr. ALFRED W. PORTER.

The author showed by means of transparencies the effect of a normal magnetic field upon the patterns which appear in developing a photographic plate which is placed between two terminals between which a sudden discharge passes. This effect is confined to the main line of discharge, and consists in this line being broadened into a band of nearly constant width; a very similar result is obtained if a blast of air sweeps across the plate instead of the magnetic field being employed. This broad band is produced by the glowing gas which surrounds the spark itself. The palm-like figures and the ramifications leading up to them do not seem to be modified by the magnetic field; and the author concludes that these figures are therefore determined by events independent of the *motion* of the changes conveyed along them, or else that the moving charges have a very large mass associated with them. In the course of the experiments the author found that in different gases at atmospheric pressure the figures obtained when the *negative* terminal is on the sensitive side of the plate are very different one from another. In air they suggest palm leaves, in carbonic dioxide they are much more feather-like, while in oxygen the ramifications are much more curvilinear than in air which forms lines which change their directions very abruptly; again, in coal-gas the figures resemble tap-roots of plants. When the positive terminal is above the plate the figures are nearly the same in all these gases; a result which is contrary to the results obtained at low pressure in vacuum-tubes. A series of transparencies was also shown showing the effects obtained in air at various pressures; very marked changes are obtained as the pressure is reduced from 76 to 10 cm.

Prof. P. T. TROUTON said the author's experiments were upon a subject which had been neglected, and might lead to fruitful results. There were a number of details visible on the plates themselves which did not show up upon the screen. It was necessary to simplify the conditions of the experiments as much as possible.

Prof. S. P. THOMPSON said the experiments opened up a fertile field of inquiry. Variations of the experiments might be tried and important results obtained. The nature of the film used obviously had something to do with the pattern produced.

Mr. A. W. PORTER, referring to Prof. Thompson's remarks, said that the figures obtained did not seem to depend upon the nature of the photographic plates used. The surrounding gas seemed to be mainly responsible for the kind of patterns produced.

Mr. ALFRED W. PORTER showed an experiment on an Anomaly in the Lagging of Thin Wires and Narrow Pipes

When thin wires and narrow pipes are covered with a lagging material it is not possible at once to say whether the covering will keep heat in or assist it to escape. There is a critical radius equal to the ratio of the conductivity of the material to the emissivity from its surface which the outer covering must exceed before it begins to act as a lagger. Although this seems to have been recognised by Prof. Bottomley and others, the results of experiments which have previously been made would seem to indicate that the effect in practice is very small indeed. The author showed a simple lecture experiment in which a large effect is obtained. A thin platinum wire (0·2 mm. radius) is coated at intervals along its length with glass of about 2 mm. outer radius. When an electric current is passed through the wire of sufficient strength to raise the uncoated portions to (say) 800° C., the coated portions do not even glow, and measurements have shown that they remain at about 100° C.; there is thus a difference of about 700° on the wrong side. A thickness of coating can be obtained which is such as to allow the same escape of heat as from a practically uncoated wire. To illustrated this, two narrow steam-pipes were shown—one coated with a single layer of asbestos paper, the other with a very thick coat. The two were equivalent in their power of preventing escape of heat. An intermediate thickness of coat would have made a *worse-lagged* pipe than either of them.

A paper "*On the Rate of Growth of Viscosity in Congealing Solutions*" was read by A. O. RANKINE, B.Sc.

The author exhibited apparatus and described experiments for the determination of the time variation at constant temperature of the viscosity of gelatin solutions of high concentration. These solutions, if quiescent, after a time possess rigidity, but continual stirring prevents its appearance and the solution remains liquid. The method of measurement, which consisted in measuring the couple on an inner cylinder when an outer concentric cylinder was rotated (the interspace being filled with the solution), secured this condition. The simple law $\eta = a - be^{-\lambda t}$ was found to hold very accurately for values of the time not too small. The values of the constants a, b, and λ are,

however, very dependent upon the temperature at which the solution is kept. On carrying the measurements to lower temperatures, it was found that a sudden rapid rise of viscosity occurred at times and in a manner over which control has not yet been obtained. It was suggested that this was analogous to the case of ordinary supersaturation. Further experiments are to be made on this point.

Dr. A. Griffiths expressed his interest in the experiments, and asked if the vigour of the stirring employed was connected in any way with the sudden change in the viscosity.

Prof. C. H. Lees pointed out that the author was not measuring the viscosity of a homogeneous liquid, but of a liquid in which lumps of gelatin were present. The sudden jump in the curve was due to a particular arrangement of these lumps.

Mr. A. O. Rankine said the rate of stirring was the same in all experiments, namely, just under two revolutions per second.

Prof. F. T. Trouton described the construction of curves to exhibit the relations which must subsist between the concentration and temperature of a solution in order that the same weight of the solute may be adsorbed per square centimetre on introducing a solid on which adsorption takes place. To such a curve the name of *isonere* has been given. When the isoneres for different weights adsorbed are compared, it is found that they consist of similar curves—that is to say, for any two isoneres the ratio of the concentration ordinates is the same for all temperatures. In addition, it is found that the saturation curve for the solution, or that showing the maximum concentration possible at each temperature, is also similar to the isoneres. Thermodynamic considerations lead to the former result provided we suppose that the latent heat of adsorption is constant for different concentrations, and to the latter if the latent heat of solution at saturation is of the same value as that of adsorption.

A paper entitled "*Note on the Recombination of Ions in Air*" was read by Dr. Phillips.

NOTICES OF BOOKS.

Komprimierte und Verflüssigte Gase. ("Compressed and Liquefied Gases"). By Dr. H. Teichmann. Halle-a-S: Wilhelm Knapp. 1908.

The use of compressed and liquefied gases commercially has during recent years been greatly extended, and a text-book dealing shortly with the subject will be welcomed. Although a gas was liquefied for the first time nearly a hundred years ago, the application of the method then adopted, and subsequently modified and improved, for the purpose of putting pure gases upon the market in a convenient form, has been the work of at most the last thirty years. Thus the author has not had to report many comparatively trifling changes in procedure and apparatus, but, on the other hand, has sometimes occasion to call attention to defects and prophesy improvements. The gas laws of Charles and Boyle are first discussed in an introductory section, and the author then passes on to the mechanical means adopted for compression, and describes the different types of compressors. The methods of preparing certain gases are next treated. In choosing the gases for consideration the author has been guided partly by their technical importance; in addition he has endeavoured to select only those which are liquefied by typical methods—passing over others for which special and very often more or less secret processes are employed. The construction of containers for compressed and liquefied gases is treated in one chapter, and the regulations in force in Germany concerning their transport and storage are also included.

Report of Recent Advances in Pharmaceutical Chemistry and Therapeutics. Vol. XXI. Darmstadt: E. Merck. 1908.

This annual report, which is sent free on application to doctors and others interested in pharmacy, gives an impartial account of advances in pharmaceutical chemistry which have been made during the year 1907. Not only are new therapeutical preparations which have come into use during the year described, but also the applications of well known drugs for fresh purposes are chronicled. Thus the book really constitutes an abstract of the pharmacopœia, and doctors will appreciate the assistance it gives them in prescribing as well as the valuable references to periodical literature.

MISCELLANEOUS.

Royal Society of Arts.—The opening meeting of the one hundred and fifty-fifth session of the Royal Society of Arts was held on Wednesday evening, the 18th inst., when an address was delivered by Sir William H. White, K.C.B., LL.D., F.R.S., Vice-President and Chairman of the Council. The following papers will be read at the ordinary meetings before Christmas:—November 25, Sir W. Martin Conway, M.A., F.S.A., F.R.G.S., "The Goldfields of Eastern Peru and Bolivia"; December 2, Eric Stuart Bruce, M.A., "Mechanical Flight"; December 9, G. Albert Smith, F.R.A.S., and Charles Urban, F.Z.S., "Kinematography in Natural Colours"; December 16, Primrose McConnell, B.Sc., F.C.S., "London Milk Supply from a Farmer's Point of View." A paper by Douglas Dewar, I.C.S., on "The Birds of India," will also be read before the Indian Section of the Society on the 10th December. There will be one course of Cantor Lectures before Christmas by Oscar Guttmann, M.Inst.C.E., F.I.C., F.C.S., on "Twenty Years Progress in Explosives." The course consists of four lectures, delivered on November 23, 30, and December 7, 14.

MEETINGS FOR THE WEEK.

Friday, 27th.—Physical, 5. "Graphic Method of dealing with Refracting Surfaces," by H. S. Allen. "Method of Determining Moments of Inertia," by (the late) W. Cassie. "Experimental Examination of Willard Gibbs's Theory of Surface Condensation regarded as the Basis of Adsorption," by W. C. M. Lewis. "Diffusion of Actinium and Thorium Emanations," by S. Russ. "Elliptic Polarisation produced by the Direct Transmission of a Plane Polarised Stream through a Plate of Quartz cut in a Direction Oblique to the Optic Axis, with a Method of Determining the Error of a Plate supposed to be perpendicular to the Axis," by J. Walker.

Monday, 30th.—Royal Society of Arts, 8. (Cantor Lecture). "Twenty Years Progress in Explosives," by O. Guttmann.

Wednesday, Dec. 2nd. - Royal Society of Arts, 8. "Mechanical Flight," by E. S. Bruce.

—— Society of Public Analysts, 8. "Gravimetric Estimation of Antimony and Tin," by E. Cahen and G. T. Morgan. "Oil from *Carapa Guianensis*," by J. Lewkowitsch. "Detection and Estimation of Formaldehyde in Milk," by H. S. Shrewsbury and A. W. Knapp. "Determination of Aldehydes in Oil of Lemon," by A. H. Bennett. "Some Analyses of Cream Cheese," by C. H. Cribb.

Thursday, 3rd.—Chemical, 8.30. "Double Salts of Potassium Iodide with Mercury Iodide and Camphor Dimercuriodide in Organic Solvents," "Action of Mercuric Iodide on Ketones in Alkaline Solution," and "Condensation of Camphor with Mercuric Iodide," by J. E. Marsh and R. de J. Fleming-Struthers. "Relation between Absorption Spectra and Chemical Constitution—Part XIII., Some Pyrones and Allied Compounds," by E. C. C. Baly, J. N. Collie, and H. E. Watson. "Organic Derivatives of Arsenic—Part I., Dicamphorylarsinic Acid," by G. T. Morgan and Miss F. M. G. Micklethwait. "Tellurium Dicyanide" and "Boron Thiocyanate," by H. E. Cocksedge.

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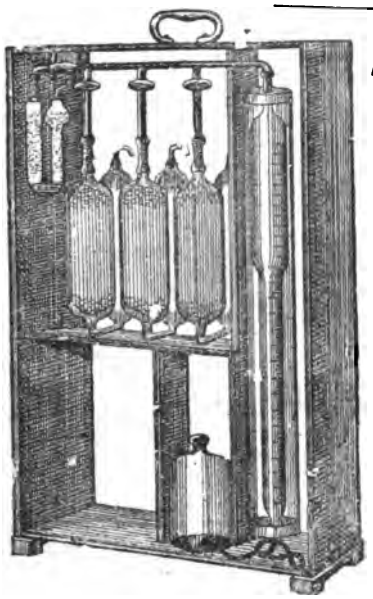

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November 26th, 1908.

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NOTICE TO ADVERTISERS.

To ensure insertion in next issue, Advertisements should be addressed to THE MANAGER, *and must reach the Office not later than* 10 *a.m. on* WEDNESDAY.

CHEMICAL SOCIETY RESEARCH FUND.

A Meeting of the Research Fund Committee will be held in DECEMBER, 1908. Applications for Grants, to be made on forms which can be obtained from the Assistant Secretary of the Society, Burlington House, W., must be received on or before MONDAY, DECEMBER 7th.

The Council wish to draw special attention to the fact that the income arising from the donation of the Worshipful Company of Goldsmiths is to be more or less especially devoted to the encouragement of research in Inorganic and Metallurgical Chemistry. Furthermore, that the income due to the sum accruing from the Perkin Memorial Fund is to be applied to investigations relating to problems connected with the Coal-tar and Allied Industries.

GOVERNMENT GRANT for SCIENTIFIC INVESTIGATIONS.—Applications for the year 1909 must be received at the offices of the Royal Society not later than the FIRST DAY OF JANUARY next, and must be made upon printed forms to be obtained from the Clerk to the Government Grant Committee, Royal Society, Burlington House, London, W.

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Vol. XCVIII., No. 2558.

THE PRESENT STATE OF OUR KNOWLEDGE OF RADIO-ACTIVITY.*

By A. DEBIERNE.

PART II.—*The Transformations of Radio-active Substances.*

In the first part of this article (*Revue Générale des Sciences*, Sept. 15, 1908, xix., p. 691; CHEMICAL NEWS, xcviii., 235 *et seq.*) we showed that four series of radio-active transformations are known. We have seen that the sequence of the different terms of each series is not a result obtained by theoretical reasoning; it is an experimental fact which has been thoroughly established in most cases.

In the modern theory of these transformations which has been worked up by Rutherford and Soddy, and which is based upon the hypothesis of atomic transmutation formerly enunciated by Pierre Curie and Mdme. Curie, each term represents a distinct chemical element which is distinguished from the ordinary chemical elements only by its short life and by the emission of a special radiation. The disappearance of the radio-active atom is the result of its transformation into another atom. The special radiation is a form of energy which accompanies this transformation. According to this theory the study of radio-activity has already led to the discovery of about twenty new elements.

However, if the chemical individuality of the heads of the series—uranium, thorium, radium—is indisputably proved by the atomic weights and the characteristic spectra, and if by analogy it may be expected that the same thing will be proved in the case of actinium, for all the other terms, as we have seen, although certain characteristics of a chemical or physical order have been observed, which enable us to distinguish and separate them (as ordinary chemical elements are separated), yet it cannot be said that these characteristics definitely prove the existence of new chemical elements. And it would not be absurd to suppose that the radio-active properties observed are due to special centres of energy which attach themselves to ordinary matter, just as electric charges or electrons attach themselves to atoms, sometimes profoundly modifying their properties, and which undergo the successive transformations. (*Cf.* the case of aqueous solutions in which the molecules are dissociated into ions). As a matter of fact, up to the present it has not been directly proved that one of the radio-active transformations previously described has ended in the disappearance of an atom, characterised by a spectrum and an atomic weight, and the appearance of a new atom characterised by a new spectrum and a new atomic weight. Even if the theory is perfectly correct this proof would be exceedingly difficult to establish, for the quantity of matter transformed in an interval of time available for experiments would be very small.

However, a certain number of facts are known which agree perfectly with the theory of an atomic transmutation; and if these facts do not directly verify the details of the above mentioned transformations they may be regarded as forming a solid experimental basis for all the theories which assume the occurrence of transformations of the atom in the phenomena of radio-activity.

I.

I shall first discuss experiments which show that radio-active substances continuously produce the gas helium. The discovery of this important fact is due to Ramsay and Soddy, who observed it with the compounds of radium.

* *Revue Générale des Sciences* 1908, xix., 730.

Giesel had discovered that radium salts decompose water into oxygen and hydrogen, and that a solution of radium continuously sets free detonating gas. Ramsay and Soddy proved in their first experiments that the gas set free by a solution of a radium salt contains a small quantity of helium. They also showed that helium is produced when radium emanation is separated by condensation in liquid air, and when this emanation is allowed to decay spontaneously. The formation of helium by radium salts is an undoubted fact. It has since been established by a great number of experimenters, including Pierre Curie and myself.

The experimental method I employed is a little different from that used by Ramsay. It consists in introducing the gases set free in a solution of radium into a tube containing different substances which absorb hydrogen, oxygen, nitrogen, and the oxides of carbon, and leave only the gases of the argon family. The gaseous residue is then driven back by means of mercury into a very fine capillary tube. A special arrangement enables the gas to be divided into two equal parts; one part is used for the spectroscopic examination, while the other is exposed to atmospheric pressure, when it forms a little bubble of gas in a capillary tube between two columns of mercury; the volume of this bubble can be determined fairly accurately. This volume is of the order of a tenth of a cubic millimetre. The spectroscopic examination showed that the helium gas is very pure. I have been able to carry out at intervals during are year a series of experiments on the same radium solution, and have thus proved that the production of helium is continuous and proportional to the time. I have also shown that the compounds of actinium exhibit the same property, and also produce helium continuously in quantities which are proportional to the time of accumulation. (The amount of helium obtained with one of my products rich in actinium was equal to that produced by about 1 centigrm. of pure radium bromide). This fact has recently been confirmed by Giesel.

Using the property of radium emanation of condensing at the temperature of liquid air, Ramsay succeeded in obtaining the emanation in a very concentrated state. He proved that it then possesses a characteristic spectrum, that it follows the ordinary laws of compression of gases, and that it gives rise to the production of helium. Finally, he showed that the volume occupied by the emanation diminishes as a function of the time. Some years ago I performed some experiments dealing with this subject, and if I did not confirm all the results stated by Ramsay and his collaborators, I did observe this diminution of volume of the emanation, which in my experiments was under atmospheric pressure. I observed that at the end of a few days the volume had become very small, being reduced to about one-twentieth of its original value. I must mention that the results of different experiments performed with the emanation do not agree very well; moreover, the experiments are very delicate, and altogether a more profound study of the question is very necessary.

The principal result of all these experiments is that radium and actinium continually produce helium, and that the production of helium is connected with the presence of active substances. I have performed many confirmatory experiments, and I have never proved the formation of helium except in presence of these substances.

The formation of helium cannot be explained in a reasonable manner, except by assuming the occurrence of an atomic transmutation.

Another very important result is due to Pierre Curie and Laborde, who measured the quantity of energy set free in radio-active transformations. The result showed that an enormous amount of energy is liberated in the phenomena of radio-activity. A grm. of radium in equilibrium with its transformation products sets free more than 100 calories per hour; *i.e.*, about 1,000,000 calories in a year. In this period of time the fraction of radium transformed can only be very small. We shall see presently that the mean duration of life of radium can be calculated upon the basis

of certain hypotheses; it would be about 3000 years. Then the total energy of transformation of 1 grm. of radium would be three thousand million calories, a quantity equivalent to that disengaged by the combustion of several tons of carbon.

The phenomenon of the liberation of heat is very easy to prove when a sufficient quantity of radium is used. It may be observed qualitatively as follows:—Two identical Dewar flasks are placed in the same receptacle in which the temperature is uniform. A tube containing radium is introduced into one of these flasks, and in the other is placed a tube containing an inactive product. A sensitive thermometer is placed in each flask. A difference of temperature between the two flasks can then be detected, that containing the radium having a temperature which is higher by some degrees. Measurements have been made with a calorimeter, and thus the result stated above has been obtained. With actinium salts I have been able to demonstrate a disengagement of heat sufficient to give a difference of some tenths of a degree between two Dewar flasks, in an experiment like that just described.

It has been shown that energy is set free in all the successive transformations which radium undergoes, and there is reason for thinking that a large part of this energy is due to the kinetic energy of the α-particles.

Whatever theories of the constitution of matter are adopted the atom must be regarded as an extremely stable system which has hitherto resisted all external agents; then it is natural to suppose that the transformation of such a stable structure would set free a much larger amount of energy than the transformation of a molecule. The production of an enormous quantity of energy in the phenomenon of radio-activity must thus be regarded as a weighty argument in favour of an atomic transformation. The fact that the energy produced is divided between the different stages of the radio-active transformations is also an argument in favour of the existence of different intermediate atoms.

The conception of an atomic transformation being agreed to, the nature of this transformation must be defined in each case.

The first condition is imposed by the laws of the transformation; it must be monatomic; that is, only one atom must be implicated at a time. (It may, however, take place between two atoms if the concentration of one is very great and practically constant). It follows that the new atoms produced can only have a mass equal or inferior to the mass of the first; the laws are unaltered when the result of the transformation is the production of several new atoms. It is difficult to imagine that the transformation of an atom lasts an appreciable time, and it is generally supposed that the transformation is instantaneous and analogous to an explosion. The different atoms which form a certain mass of matter are successively transformed with a frequency which depends upon their nature.

The production of a special radiation, which consists in a projection of particles of different kinds, agrees equally well with the hypothesis of an explosion.

Very definite hypotheses concerning the nature of the transformation have been formulated by Rutherford. In the first place when a radio-active atom is transformed only one new radio-active atom is produced. For instance, one atom of radium which gives one atom of emanation gives one atom of radium A, &c. To explain the formation of helium, Rutherford and Soddy have supposed that the α-rays are formed by the projection of positively charged helium atoms. To make this hypothesis agree with the value of e/m for the α-rays, Rutherford has assumed that the charge e is double the charge on an element in electrical phenomena. He has also supposed that in each transformation with α-rays only one α-particle is emitted; that is, only one helium atom for each radio-active atom which undergoes transformation. When the transformation takes place without the emission of α-rays the mass of the atom does not undergo any variation in the transformation. Hence it follows that the different terms of a series must have atomic weights which do not differ much from one another.

The atomic weight of radium being 226·5, that of the emanation will be 224·5, that of radium A 218·5, radium B 214·5, radium C 214·5, since radium B does not emit α-rays, radium D 210·5, radium E 210·5, radium F 210·5, and the last unknown term 206·5.

This last atomic weight, which is very nearly that of lead, led Rutherford and Boltwood to suppose that the result of the transmutation of radium is lead. Boltwood has endeavoured to support this view by instancing the lead contained in radio-active minerals; but according to more recent researches it appears that some radium minerals do not contain lead. The formation of lead from radium is thus as yet purely hypothetical.

The results of experimental determinations of the atomic weights of the gaseous emanations do not agree with the numbers given above.

We know that the coefficient of diffusion of a gas is approximately inversely proportional to the square root of its molecular weight. The concentration of an emanation being easily determined relatively, it is possible to determine the coefficients of diffusion of the different emanations in different gases.

The measurements are not very accurate for the emanations of thorium and radium, but the numbers point to a molecular weight equal to about 100. With actinium I have been able to get an accurate result, and have found that the molecular weight is 70.

If it is supposed that the emanations are monatomic gases of the argon family, which is in agreement with the fact that they cannot be made to enter into combination, the molecular weight is the same as the atomic weight, whereas the number is at least twice as small as that given by the above theory. (If the emanations were diatomic the atomic weights would be twice as small again). These numbers, obtained by diffusion experiments, are founded upon an experimental basis which seems to be correct, and I think that there is reason for believing that in the transformation of an atom of radium two atoms of emanation are formed. This makes no change in the law of transformation. It also seems possible that phenomena of agglomeration occur in the other terms of the series. This agglomeration might have no influence on the true laws of radio-active transformations.

In all theories of atomic transmutation we are led to the conclusion that the active substances disappear gradually with the time. This phenomenon must therefore occur with the heads of the series uranium, thorium, radium, and actinium. No disappearance has been proved directly by experiment; this shows at any rate that only a very small fraction of the total mass is transformed at the end of several years. Nevertheless, attempts have been made to calculate indirectly the hypothetical law of decay and the mean duration of life of these active substances. Some of the above hypotheses are used in the calculation.

Rutherford based one of his calculations on the magnitude of the positive charge expelled in the form of α-particles.

By assuming that only one α-particle is expelled when a radium atom disappears, and that this particle has an electric charge equal to twice the charge on an element, and by determining experimentally the electric charge expelled in a given time for a known quantity of radium, the data are obtained for calculating the proportion of atoms which disappears per second and the mean duration of life of a radium atom. A similar but less direct calculation has been made for uranium and thorium.

Moreover, in different experiments Ramsay has obtained the emanation in a very concentrated state, and has measured the volume of emanation produced in a given time. Adopting the hypothesis that an atom of emanation is produced by the disappearance of an atom of radium, the mean duration of life of radium can also be calculated. This period could also be obtained by observing the volume of helium yielded, employing the above hypotheses on the formation of helium.

The numbers thus obtained, based on hypotheses which have not yet been established, and upon experiments which are by no means accurate, vary within fairly wide limits. The mean duration of life would be between 250 and 3000 years, that of uranium would be of the order of 5×10^9 years, and that of thorium of 10^{10} years.

(To be continued).

ON HIGH TENSION BRONZES.

By SERGIUS KERN, M.E., St. Petersburg.

HIGH tension bronzes—as, for instance, manganese bronze—deserve more attention than they usually receive from engineers. Apart from their physical qualities (tensile strength and elongation), which come near to those of steel castings, and the keeping of these qualities with small diminution at temperatures of high-pressure marine boilers (which the ordinary gun-metal fails to do), the high tension bronzes, containing about 60 per cent copper, 38 per cent zinc, and certain quantities of iron and manganese, have none or only small quantities of tin. This metal may be considered a speciality for liquation processes.

Such a nuisance as liquation we often observe in gun-metal castings for different fittings (valves, &c.) for high-pressure boilers. This is a great evil, as it gives rise to a heavy loss in ready machined fittings, which before delivery must stand serious and heavy hydraulic testing (very often 600 lbs. to the square inch). The cause of failures is chiefly due to liquation effects, which were not so harmful when small pressures in testing were applied (say, 90 lbs. per square inch), when marine boilers worked at low pressures. At those times gun-metal castings satisfied the demands of engineers.

Some years ago we made many trials and witnessed many experiments with Parsons' manganese bronze, and what struck us was the absence of liquation, speaking practically, in high tension bronzes, and that is the reason why we always insisted that such bronzes, apart from their physical qualities, must be more widely used instead of the old gun-metal.

In conclusion, we may add that small defects in high-tension bronzes when tried under hydraulic pressure, may be easily and safely chased by hammering. Such a method for gun-metal castings is out of the question.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 265).

Confirmatory Experiments and References (continued).

Solubility of Uranyl Vanadate in Sodium Hydrogen Carbonate Solutions.—Solutions containing 100 mgrms. U as $UO_2(NO_3)_2$ and 60 mgrms. V as Na_3VO_4 were mixed, a few drops HNO_3 (1·20) were added to dissolve the precipitate, the mixture was diluted to 100 cc., 2 grms. $NaHCO_3$ were added, and the mixture was digested in a closed bottle at 95° for half an hour; no precipitate separated. The mixture was cooled down, 40 mgrms. V as Na_3VO_4 were added, making 100 mgrms. in all, and the mixture was again heated at 95° for half an hour; no precipitate separated in the hot solution, nor in the cold on standing several days. The experiment was repeated with 100 mgrms. U and 100 mgrms. V, except that the mixture was first treated with NaOH and H_2O_2 by P. 52, and that 1·5 grms. $NaHCO_3$ were used instead of 2 grms.; the result was nearly the same. 100 mgrms. U as $UO_2(NO_3)_2$, 100 mgrms. V as Na_3VO_4, and 3 grms. $NaNO_3$ were treated by *P. 58*a*, 1 grm. excess of $NaHCO_3$ being added; a precipitate formed on adding $NaHCO_3$, and a considerable one remained after heating the mixture at 100° in a closed bottle for thirty minutes. The precipitate and filtrate were analysed for uranium and vanadium by *P. 58*c*—*e*: the filtrate was found to contain 35 to 40 mgrms. of uranium, and all but 10 or 15 mgrms. of the vanadium.

The last experiment was repeated except that the mixture was warmed in a flask at 95° (without allowing it to boil) for thirty minutes; the precipitate that remained was smaller, and contained only 10 to 20 mgrms. U and 2 to 5 mgrms. V. The last experiment was repeated except that the mixture was boiled for one minute in an open flask; the precipitate dissolved completely, and no precipitate formed on cooling even in twenty-four hours, showing that uranyl vanadate is more soluble in slightly alkaline carbonate solutions than in those from which the CO_2 is prevented from escaping.

An acid solution containing 50 mgrms. V as Na_3VO_4, and 50 mgrms. U as $UO_2(NO_3)_2$ was diluted to 100 cc. and neutralised by adding a little powdered $NaHCO_3$. Then 0·5 grm. $NaHCO_3$ was added, and the mixture was heated in a pressure bottle at 95° for half an hour; on heating a large precipitate remained. To a solution containing 100 mgrms. V as Na_3VO_4, 20 mgrms. U as $UO_2(NO_3)_2$ and a known excess of HNO_3 (1·20) in 50 cc. (instead of 100 cc.) was added just sufficient $NaHCO_3$ to leave 1 grm. undecomposed $NaHCO_3$. The mixture was heated in a closed bottle at 95° for half an hour; no precipitate separated. To this solution after cooling, was added 20 mgrms. more U and the mixture was again heated at 95°; a large precipitate separated in the cold and remained on heating. The experiment was repeated with 100 mgrms. U and 10 mgrms. V: no precipitate separated. 10 mgrms. more V were added and the heating repeated; a large precipitate separated in the cold and remained on heating. The last experiment was repeated with 100 mgrms. V and 100 mgrms. U, and the precipitate and filtrate were analysed for uranium and vanadium by *P. 58*c* to *e*; the filtrate was found to contain 20 or 30 mgrms. of uranium and all but a few mgrms. of the vanadium.

Non-Precipitation of Uranyl Chromate, Phosphate, and Oxalate in the $NaHCO_3$ *Procedure.*—To a solution containing 100 mgrms. U as $UO_2(NO_3)_2$ 200 mgrms. Cr as K_2CrO_4, and a known (small) amount of HNO_3 (1·20) in 100 cc. was added enough $NaHCO_3$ to leave 1 grm. of the undecomposed carbonate; the mixture was heated in a closed bottle at 95° for half an hour; no precipitate separated. A solution containing 100 mgrms. U as $UO_2(NO_3)_2$ and 100 mgrms. PO_4 as $NaHPO_4$ was treated by P. 52 and then by *P. 58*a*; no precipitate separated in the $NaHCO_3$ procedure. After cooling an additional 250 mgrms. portion of PO_4 was added, and the heating repeated; no precipitate separated. The experiment was repeated with 100 mgrms. V as Na_3VO_4 and 200 mgrms. oxalic acid; no precipitate separated.

*P. 58*a*, N. 4: *Precipitation of Aluminium, Zinc, and Beryllium in Hot Sodium Hydrogen Carbonate Solutions in Open Flasks.*—2 mgrms. Al as nitrate and 3 grms. $NaNO_3$ were dissolved in 100 cc. water; a little $NaHCO_3$ was added until the mixture after shaking just ceased to react acid to litmus-paper; 1 grm. more was added; the mixture was heated to about 90°, and digested at this temperature in a covered flask on a water-bath for half an hour; the precipitate slowly dissolved, except a few small flakes

* From the *Journal of the American Chemical Society*, XXX., No.4.

The mixture was filtered while hot, and the filtrate tested for aluminium by acidifying with acetic acid, adding Na_2HPO_4 and heating to boiling; almost all the aluminium was found in the filtrate. This experiment was repeated twice with 2 mgrms. and with 1 mgrm.; the results were the same. The experiment was repeated twice with 20 mgrms. Al; the filtrate contained less than 1 mgrm. Al.

The experiments with 2 and 20 mgrms. were repeated, except that CO_2 gas was passed through the mixture; the results were nearly the same, except that the filtrate may have contained somewhat more aluminium, showing that there is no advantage in using CO_2 gas.

The experiment with 20 mgrms. Al was repeated except that the mixture was boiled for one minute; the filtrate contained 3 or 4 mgrms. Al. The experiment was repeated except that the mixture was allowed to cool before filtering; the filtrate contained about 1 mgrm. Al. The experiment was repeated with 4 mgrms. Al; the precipitate disappeared completely on boiling, but a precipitate separated out on cooling. The experiment was repeated with 6 mgrms. Al; a very small precipitate remained on boiling one minute.

1 mgrm. Zn as nitrate and 2 grms. $NaNO_3$ were dissolved in 100 cc., 1 grm. excess $NaHCO_3$ was added, and the mixture boiled in a flask for one minute; the precipitate did not dissolve and was proved to contain zinc by P. 53—57. The experiment was repeated with 20 mgrms. Zn; the filtrate being tested for zinc by acidifying with HNO_3, boiling, adding NH_4OH, and passing in H_2S; no precipitate formed, showing that the zinc was completely precipitated.

10 mgrms. Be as chloride and 2 grms. $NaNO_3$ were dissolved in 100 cc. water, $NaHCO_3$ was added until the mixture after shaking just ceased to react acid to litmus-paper; 1 grm. more was added; the mixture was boiled for one minute, and filtered while hot; the filtrate was acidified with HNO_3, evaporated to about 15 cc., and tested for beryllium by adding NH_4OH; the filtrate contained only about 0·1 mgrm. Be. The experiment was repeated except that the mixture was allowed to cool before filtering; the filtrate contained about 0·3 mgrm. Be. The experiment was repeated with 1 mgrm. Be; the precipitate remained on boiling for one minute, and did not disappear on standing in the cold for several hours.

These experiments were repeated with solutions containing from 1·5 to 5 grms. $NaHCO_3$ in 100 cc. of solution and varying quantities of beryllium; in a 1·5 per cent $NaHCO_3$ with 1 mgrm. Be little or no precipitate remained on boiling one minute; in a 2 per cent solution no precipitate remained with 2 mgrms. Be, but one remained with 4 mgrms.; in a 3 per cent solution 40 mgrms. Be dissolved almost completely, but 50 mgrms. gave a large precipitate. A comparison of these results with those obtained in a similar series of experiments in closed bottles (C. E., N. 3, above) shows that considerably more beryllium dissolves in concentrated $NaHCO_3$ solutions when the CO_2 is allowed to escape.

*P. 58*a*, N. 5: *Precipitation of Uranium with Aluminium or Beryllium in Hot Dilute Sodium Hydrogen Carbonate Solutions.*—See T. A., No. 171 to 175. In an analysis with 200 mgrms. Al and 10 mgrms. U, starting at *P. 58*a*, 1 or 2 mgrms. U were found in *P. 58*c*—*d*.

*P. 58*a*, N. 6: *Dissociation Relations of Carbonic Acid.*—See McCoy (*Am. Chem. Journ.*, 1903, xxix., 437).

Dissociation Relations of Chromic Acid.—See Spitalsky (*Zeit. Anorg. Chem.*, 1907, liv., 265) and Sherrill (*Journ. Am. Chem. Soc.*, 1907, xxix., 1641). The work of the latter shows that H_2CrO_4 dissociates in steps, first into HCr_4O^- and then into $CrO_4^=$, that the $HCrO_4^-$ is a very weak acid, and that the latter even in dilute solution is converted by dehydration in large measure, but by no means wholly, into dichromate-ion ($Cr_2O_7^=$).

(To be continued).

ON SCANDIUM.*

By Sir WILLIAM CROOKES, D.Sc., F.R.S.

SCANDIA is one of the rarest and least known of the recognised rare earths. It was discovered in 1879 by Nilson, who separated it, with ytterbia, from erbia extracted from euxenite and gadolinite. Nilson made an incomplete chemical examination of some of its compounds, but owing to the inadequate amount of material at his disposal he did not at first entirely separate it from ytterbia. Later in the same year Cleve extracted scandia from gadolinite, yttrotitanite, and keilhauite, and described the scandium sulphate, double sulphates, nitrate, oxalate, double oxalates, selenate, acetate, formate, oxide, and hydrate, and gave some of the chief reactions of the new body.

Cleve, working on gadolinite, found that it contained only from 0·002 to 0·003 per cent of scandium, while keilhauite yielded only about 0·005 per cent. He gave results from which he deduced an atomic weight of about 45. Cleve noticed that scandium almost exactly corresponded to the description given by Mendeleeff of his hypothetical element "ekaboron," of atomic weight 44. More recently, in 1880, Nilson, working on somewhat larger quantities of scandia, described and analysed the nitrate, sulphate, selenate, oxalate, and the potassium double sulphate. He found the atomic weight to be 44·03, the mean of four separate and closely concordant determinations. Taking Nilson's data, and re-calculating from them the atomic weight of scandium, using the most recent figures for oxygen and sulphur, I find his atomic weight to be almost exactly 44·1—a figure I have used in the following paper.

In the course of my twenty years' work on the fractionation of the rare earths I have repeatedly tested my products by examining their photographed spectra, using the dominant lines of the various elements as tests for their presence. Scandium has an extremely characteristic group of lines in its spectrum, situated between wave-length 3535·864 and wave length 3651·983, the strongest being at 3613·984, midway between two strong iron lines. By using a part of the spectrum in which this line occupies the centre of the photograph, it is easy to recognise scandium. Detecting the dominant line, the presence of scandium can be verified by reference to the other lines of the group. (See Plate).

Scandium I found in some of my fractions, but only in small quantities. A few years ago I commenced an examination of all the obtainable rare-earth minerals, in order to see if any of them showed more than a trace of scandium. The minerals examined are given in next col.

Of these minerals scandium was detected in auerlite, cerite, cryptolite, keilhauite, koppite, mosandrite, orangite, orthite, pyrochlore, thorianite, thorite, urdite, and wiikite. But while the other minerals contained less than 0·01 per cent of scandium, wiikite was found to contain more than one hundred times that amount.

Wiikite is a black amorphous mineral found with monazite in a felspar quarry at Impilaks, Lake Ladoga, Finland. It is named after Professor Wiik, of Helsingfors (*Min. Mag.*, vol. xiii., p. 379).

I have compared wiikite with a large number of typical rare-earth minerals, and in appearance I find it most resembles yttrotantalite. A thin section under the microscope looks like solidified mud. It is quite amorphous, breaks with a conchoidal fracture, shows no crystalline structure, and has no action on polarised light.

Wiikite is somewhat radio-active. A fragment was laid on a sheet of sensitive film—opaque black paper intervening—for ninety-five hours. Subsequent development revealed a good impression, about equal in strength to what would be given by pitchblende in twenty-four hours. The image was not uniform, and at one point radiation had spread from the mineral over the adjacent part of the sensitive film.

* A Paper read before the Royal Society, April 30, 1908.

Æschynite	Polycrase
Allanite	Pyrochlore
Alvite	Rhabdophane
Auerlite	Samarskite
Baddelite (Ceylon)	Scheelite (Bohemia)
Bastnasite	Scheelite (New Zealand)
Brüggerite	Schorlomite
Cerite	Sipylite
Clevite	Tantalite
Columbite	Thalenite
Cryptolite	Thorianite
Eudialite	Thorite
Euxenite	Thorogummite
Fergusonite (Ceylon)	Tscheffkinite
Fergusonite (Ytterby)	Tysonite
Fluocerite	Urdite
Gadolinite	Wiikite
Hielmite	Xenotime
Homolite	Yttergarnet
Keilhauite	Yttrialite
Knopite	Yttrocerite
Koppite	Yttrogummite
Lanthanite	Yttrotantalite
Monazite	Yttrotitanite
Mosandrite	Zirkelite (Ceylon, sp. gr. 5·0)
Orangite	Zirkelite (Ceylon, sp. gr. 4·42)
Orthite	

The specific gravity of wiikite is 4·85. Its hardness is 6. It is infusible before the blowpipe. It is imperfectly attacked by strong mineral acids and breaks up easily when fused with potassium bisulphate. Heated to full redness in a silica tube it gives off helium, water, and a distinct amount of sulphuretted hydrogen, followed by a white sublimate. The mineral begins to crack at a temperature a little below redness, and, at the approach of redness, gas is evolved with almost explosive violence, the mineral breaking and flying about the tube. A fragment so treated examined under the microscope shows a surface covered with glistening points. With a high power these points are resolved into a mass of minute cubes, curiously regular in form and appearance. Heating drives off 5·83 per cent of its weight; 5·82 of the loss is water and acid vapour, the difference, 0·01 per cent, consists chiefly of helium, with a little hydrogen, carbon dioxide, and a mere trace of neon. The average amount of gas collected from 100 grs. of the mineral was 15·6 cc. Sir James Dewar kindly passed the gases for me over hot oxide of copper and then through charcoal immersed in liquid air. This should absorb and retain everything but helium and the trace of neon. The gas thus purified, after exhaustion to the most luminous point, was sealed in a double-bulb silica tube. The spectrum, visual and photographed, was that of pure helium with a scarcely perceptible trace of neon.

Containing so many bodies, the exact separation of which one from the other is not known, it is at present impossible to give an accurate and complete analysis of wiikite. Moreover, I find the composition varies somewhat in different samples of the mineral. The following is considered a fair approximation of the composition of a typical specimen :—

Tantalic acid with some niobic acid	15·91
Titanic acid and zirconia	23·36
Earths of the cerium group..	2·55
Earths of the yttrium group.	7·64
Scandia	1·17
Thoria	5·51
Ferrous oxide..	15·52
Uranic oxide..	3·56
Silica	16·98
Water and gases	5·83
Calcium, manganese, tin, sulphur, &c., unestimated	1·97
	100·00

The percentage of scandia is too low. Owing to the difficulty of separating it completely from the accompanying earths and metallic acids, the amount given is only that which has been obtained in an approximately pure state.

I find the following the best process to extract scandia from wiikite :—The ground mineral is passed through an 80-mesh sieve, then mixed with five times its weight of powdered potassium bisulphate and fused in a clay crucible. At first much frothing occurs, due to the escape of permanent gases and aqueous vapour; this ebullition can be abated by stirring with an iron rod. When in quiet fusion, the heat is raised to full redness for ten minutes and the liquid mass poured on an iron plate. When cold, the melt is finely ground, mixed with water in the proportion of 1 kilo. to 5 litres, and mechanically agitated for about twelve hours. It is then thrown on a linen filter and well washed. This treatment extracts most of the rare earths, together with a little titanic, niobic, and tantalic acids and zirconia, but the bulk of these bodies is left behind.

The solution is supersaturated with ammonia and well boiled; rhis treatment precipitates all the earths, together with iron, titanic acid, zirconia, &c. The precipitate of crude earths is filtered and well washed.

The precipitated crude earths are converted into oxalates by heating the pasty precipitate in a dish with crystals of oxalic acid added gradually, excess being avoided as much as possible. When cold, the oxalates are filtered off, and the iron, &c., in the filtrate is precipitated with ammonia. As scandium oxalate is slightly soluble in water, the iron precipitate will contain a certain proportion, which must be kept for subsequent working up.

The washed oxalates are dried and ignited; the resulting earths form about 17 per cent of the mineral and contain about 7 per cent of scandia.

The crude oxides are converted into sulphates by heating with sulphuric acid, the excess of acid being driven off by heat—not using too high a temperature. The dry sulphates are agitated with plenty of water, and filtered from a little insoluble residue of metallic acids. Ammonia in excess is then added to the filtrate, and the precipitated oxides are washed, once more converted into oxalates in the manner already described, and the oxalates again washed, dried, and ignited. The ignited oxides are boiled in dilute nitric acid, filtered from a little insoluble matter, and evaporated to dryness. These somewhat complicated operations are to insure the removal of sulphuric and metallic acids, the presence of which interferes with subsequent fractionation.

The earthy nitrates rich in scandium are mixed with an equal bulk of potassium nitrate and fused in large porcelain crucibles with constant stirring until the mass has the appearance of cream—fluid when hot, solidifying on cooling to a white enamel-like mass. On boiling this mass in water, a dense white basic nitrate separates and undecomposed nitrates are left in solution. Scandium and ytterbium, forming easily decomposed nitrates, concentrate in the insoluble end—the "heads"—while the yttrium and cerium earths, whose nitrates are less easily decomposed by heat, are left in the soluble end—the "tails." Fractionation on these lines must now be continued until spectrum photographs show the scandium to be pure. It is not difficult to obtain scandia free from all earths present except ytterbia and yttria; but the final elimination of pure scandia from these earths is a matter demanding much time and patience.

This easy decomposition of the nitrate by heat makes it possible by a systematic series of fractionations to separate scandium from most of the other associated elements. The worst impurity is ytterbium, the nitrate of which is almost as easily decomposed. Fortunately a strong dominant line of the ytterbium spectrum, wave-length 3694·344, occurs at a vacant part of the scandium spectrum, and near the characteristic group of scandium. A sample of scandia is not considered satisfactory if the least trace of this line is seen on an over-exposed spectrum of scandium, and if the atomic weight is higher than 44·1. The atomic

weight of ytterbium being 173, a very little of it as an impurity raises the atomic weight of scandium.

SCANDIUM HYDROXIDE,

$Sc_2O_3,3H_2O = Sc(OH)_3$.

On adding caustic alkali to a solution of a scandium salt the hydroxide comes down as a bulky gelatinous precipitate. It is insoluble in excess of precipitant. In appearance it resembles yttrium hydroxide. It dries in the air at ordinary temperatures to a hard porcelain-like mass and has the composition given above.

Analysis.

5·289 grs. lost on ignition 1·533 grs. of water.

	Theory.		Experiment.
Sc_2O_3	136·200	71·59	71·02
$3H_2O$	54·048	28·41	28·98
	190·248	100·00	100·00

The hydroxide is a very weak base, with a marked tendency to form basic salts. It dissolves readily in dilute acids, forming salts which have a sweet astringent taste. The salts show no absorption spectra.

After ignition, anhydrous scandia, Sc_2O_3, is difficultly soluble in dilute acids in the cold, rather more easily soluble when warmed, and readily soluble in hot strong mineral acids. Strong sulphuric acid poured on ignited scandia attacks it with considerable evolution of heat.

SCANDIUM CARBONATE,

$Sc_2(CO_3)_3,12H_2O$,

comes down as a bulky white precipitate on adding ammonium or sodium carbonate to a soluble salt of scandium. It is almost insoluble in a cold dilute solution of ammonium carbonate, but is moderately soluble in a hot and strong solution of this salt, and as in the same circumstances yttrium carbonate is much less soluble, this difference may be utilised as a basis for a method of separating scandium and yttrium by fractionation.

Scandium carbonate holds carbonic acid very weakly, and concordant analyses are not easy to get. Drying at 100° drives off water and some of the carbon dioxide, leaving a basic carbonate. When freshly precipitated and dried in a current of air at the ordinary temperature, the following formula best agrees with the figures obtained :—

$Sc_2(CO_3)_3,12H_2O$.

At a temperature of 100° this salt loses carbon dioxide and water, and becomes a basic salt of uncertain composition.

Strong ignition drives off all water and carbon dioxide, leaving Sc_2O_3.

HYDRATED SCANDIUM CHLORIDE,

$Sc_2Cl_6,12H_2O$.

The hydrated salt is prepared by dissolving the earth in hydrochloric acid and evaporating on a water-bath. It crystallises from a strong solution as a felt-like mass of fine white needles. The crystals should be pressed many times between filter-paper frequently renewed, and put to dry for a few hours in a desiccator over sulphuric acid. They are somewhat deliquescent, but not so much so as the nitrate, and are readily soluble in alcohol. They have the following composition :—

$Sc_2Cl_6,12H_2O = Sc_2O_3,6HCl,9H_2O$.

Analysis.

1. 6·087 grs. of the pressed chloride left on ignition 1·620 grs. of scandia.
2. 5·960 grs. of the pressed chloride lost 1·836 grs. after remaining in the water-oven at 100° for six hours.

	Theory.		Experiment.
			1 and 2.
Sc_2O_3	136·200	26·34	26·61
6HCl	218·748	42·30	42·58
$9H_2O$	162·144	31·36	30·81
	517·092	100·00	100·00

When heated for six hours to a temperature of 100°, the 9 molecules of water go off, leaving a clear, colourless, and oily liquid, which on cooling solidifies to a white crystalline mass of the composition

$Sc_2Cl_6,3H_2O = Sc_2O_3,6HCl$.

When this salt is gradually heated to redness it gives off 6 molecules of hydrochloric acid with much intumescence, leaving a bulky residue of scandia.

Analysis.

1. 4·235 grs. on ignition left 1·620 grs. of scandia, = 1·049 grs. of scandium.
2. 4·124 grs. were dissolved in water and the scandia precipitated by ammonia. The scandia obtained weighed 1·592 grs., = 1·031 grs. of scandium.

	Theory.		Experiment.	
			1.	2.
2Sc	88·200	24·85	24·77	25·00
6Cl .. .. }	266·748	75·15	75·23	75·00
$3H_2O$ . . }				
	354·948	100·00	100·00	100·00

HYDRATED SCANDIUM BROMIDE,

$Sc_2Br_6,12H_2O$.

Scandium hydroxide or carbonate dissolves easily in hydrobromic acid, and the acid solution evaporated on the water-bath deposits small rhombic crystals, coloured slightly brown by free bromine and very deliquescent. These crystals were collected on filter-paper and dried by pressure between folds of paper frequently renewed. They were then put into a silica weighing bottle, and dried in a desiccator over sulphuric acid.

Analysis.

1. 29·753 grs. of the hydrated bromide gave on ignition 5·068 grs. of scandia.
2. 29·753 grs. of the hydrated bromide lost at 120° 6·207 grs. of water.

	Theory.		Experiment.
			1 and 2.
Sc_2O_3	136·200	17·37	17·03
6HBr	485·808	61·95	62·11
$9H_2O$	162·144	20·62	20·86
	784·152	100·00	100·00

When the hydrated bromide is heated in an air-bath to 120° it loses 9 molecules of water and becomes—

$Sc_2Br_6,3H_2O = Sc_2O_3,6HBr$.

Analysis.

23·546 grs. of the dried bromide left on ignition 5·068 grs. of scandia.

	Theory.		Experiment.
Sc_2O_3	136·200	21·89	21·53
6HBr	485·808	78·11	78·47
	622·008	100·00	100·00

SCANDIUM FLUORIDE,

Sc_2F_6.

Ignited scandia is attacked by aqueous hydrofluoric acid, forming a semi-transparent gum-like mass, which on boiling

changes to a fine white precipitate. This is difficult to filter, and it has great tendency to run through.

When hydrofluoric acid is added to a solution of scandium nitrate or sulphate, a white precipitate comes down. On boiling it becomes milky, resembling the above-described precipitate.

The fluoride dried over sulphuric acid is anhydrous. It slightly loses weight at a red heat, and at a yellow heat it frits together, fusing with difficulty before the blowpipe.

Strong sulphuric acid imperfectly decomposes scandium fluoride. It is completely decomposed by fusion with potassium bisulphate; the melt dissolves in acidulated water, and the solution mixed with ammonia deposits scandium hydroxide.

Analysis.

8·955 grs. of scandium fluoride, decomposed by fusion with potassium bisulphate and precipitated by ammonia, gave 6·0365 grs. of scandia, = 3·9091 grs. of scandium.

		Theory.	Experiment.
Sc_2	88·2	43·62	43·65
F_6	114·0	56·38	56·35
	202 2	100·00	100·00

Scandium Chlorate.

Aqueous chloric acid saturated with scandium hydroxide forms at first a clear colourless liquid; evaporation on the water-bath decomposes it, with evolution of chlorine. On further evaporation, crystals like long needles form on cooling, and the mother-liquor becomes a gummy amorphous mass in which, after standing, appear long needle-shaped crystals. These crystals probably are the perchlorate.

Scandium Perchlorate.

Aqueous perchloric acid easily dissolves scandium hydroxide or carbonate, and the solution evaporated over the water-bath separates into a felt-like mass of rhombic needle-shaped crystals.

Scandium Bromate.

Bromic acid readily dissolves scandium hydroxide, but on evaporating the solution it decomposes, leaving an amorphous gummy mass without crystalline appearance.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 19th, 1908.

Sir William Ramsay, K.C.B., F.R.S., President, in the Chair.

The President announced that the Society had become indebted to Prof. J. Emerson Reynolds, F.R.S., for presenting a bust of Michael Faraday.

Certificates were read for the first time in favour of Messrs. Arthur Clayton, B.Sc., 35, Sisters Avenue, Clapham Common, S.W.; John Charles Jesser Coope, Bulawayo, Rhodesia, S. Africa; Arthur Gordon Francis, B.Sc., 50, Whitbread Road, Brockley, S.E.; Ross Aiken Gortner, M.A., B.Sc., Lincoln, Nebraska, U.S.A.; Frank Higson, Ivy Bank, Castle Street, Haulgh, Bolton; James Henry Hoseason, Alderside, Timperley, Cheshire; Ralph Hough, 14, Longford Place, Victoria Park, Manchester; Percy May, 126, Cazenove Road, Stamford Hill, N.; Frank George Pope, 28, Alcester Crescent, Upper Clapton, N.E.; Philip John Sageman, 160, Brecknock Road, N.

Certificates have been authorised by the Council for presentation to ballot under By-law I. (3), in favour of Messrs. Thomas Boles Allen, M.A., Medical Faculty, McGill University, Montreal; Maung Ba, B.A., Rangoon College, Rangoon; Prince Mom Chow Bhunsri, Saranrome Palace, Bangkok, Siam; Maung San U, Government Laboratory, Rangoon; Herbert M. Shilstone, B.Sc., 410, South Front Street, New Orleans, U.S.A.; Rev. John Watt, M.A., Scottish Churches College, Calcutta.

Of the following papers, those marked * were read:—

*238. "*Meteloidine; a New Solanaceous Alkaloid.*" By Frank Lee Pyman and William Colebrook Reynolds.

Datura meteloides yields 0·4 per cent of total alkaloid, from which 0·13 per cent of hyoscine, 0·03 per cent of atropine, and 0·07 per cent of a new alkaloid, *meteloidine*, have been obtained.

Meteloidine, $C_{13}H_{21}O_4N$, crystallises in tabular needles, melting at 141—142°. It forms crystalline salts, of which the *hydrobromide*, $C_{13}H_{21}O_4N,HBr,2H_2O$ (needles, m. p. 250°), *aurichloride*, $C_{13}H_{21}O_4N,HAuCl_4,\frac{1}{2}H_2O$ (hexagonal plates, m. p. 149—150°), and *picrate* (hexagonal plates, m. p. 177—180°) have been prepared. It is optically inactive, and has no marked physiological action.

It is readily hydrolysed by hot aqueous barium hydroxide, with the formation of tiglic acid and a new base, *teloidine*, according to the equation $C_{13}H_{21}O_4N + H_2O = C_5H_8O_2 + C_8H_{15}O_3N$.

Teloidine crystallises from moist acetone in needles, which contain one molecule of water of crystallisation, and, after drying at 120°, melts at 168—169°. It forms crystalline salts, of which the *hydrochloride*, $C_8H_{15}O_3N,HCl$ (prisms, m. p. above 300°), *hydrobromide*, $C_8H_{15}O_3N,HBr$ (hexagonal plates, m. p. 295°), and *aurichloride*, $C_8H_{15}O_3N,HAuCl_4,\frac{1}{2}H_2O$ (hexagonal plates, m. p. 225) have been characterised.

Attention was drawn to the similarity of the properties of teloidine with those of tropine and oscine.

*239. "*The Affinity of certain Alkaloids for Hydrochloric Acid.*" By Victor Herbert Veley.

Determinations have been made of the affinity values of the nitrogen atoms or amino-groups of certain alkaloids, which, although containing more than one atomic proportion of nitrogen, yet form stable combinations with one molecular proportion of hydrogen chloride, the dihydrochlorides being produced only under special conditions, or not, as yet, having been obtained. It was shown by the application of the methyl-orange method that in all cases the affinity values of these amino-groups are of a widely different order.

In the case of the cinchona alkaloids, the affinity value of one amino-group is rather less than that of a hydropyridine, whilst that of the other is rather lower than that of quinoline. These results are in accordance with the accepted constitution of these alkaloids and the thermoneutrality values obtained by the addition of one and two molecular proportions of hydrogen chloride respectively.

In the case of pilocarpine, the value of one grouping is intermediate between that of ammonia and the limit value of the methyl-orange method, whilst that of the other is equal to those of true amphoteric electrolytes.

A methylglyoxaline was examined on account of the relationship of pilocarpine to these compounds; the value of the two amino-groups and its general behaviour correspond with a diamino-base of the type of hydrazine.

Of the *Nux Vomica* alkaloids, strychnine and brucine were examined; in both cases, the affinity value of one amino-group was beyond the limit value of the methyl-orange method, whilst that of the other corresponded with the betaines, which is in accordance with the views held regarding their constitution.

Discussion.

Dr. Veley, in reply to Dr. Jowett, stated that so far as results had been obtained it appeared probable that if an alkaloid was constituted of two residues, either or both

containing an amino-group, the affinity value of the one was affected by that of the other.

This had been observed more especially in the case of tropine, the parent base of the atropine and cocaine alkaloids, the results for which it was hoped shortly to communicate to the Society.

*240. "*The Relation between Absorption Spectra and Chemical Constitution.* Part XII. *Some Amino-aldehydes and -ketones of the Aromatic Series.*" By EDWARD CHARLES CYRIL BALY and EFFIE GWENDOLINE MARSDEN.

The absorption spectra of *o*- and *p*-aminobenzaldehyde and the three isomeric aminoacetophenones have been observed; they all exhibit a single broad absorption band which is within the visible region of the spectrum, so that the substances are visibly coloured. The addition of a small quantity of alcoholic hydrochloric acid to the solution of each of the compounds causes a considerable deepening of the colour, which is due to the formation of a new absorption band nearer to the red than the principal band referred to above. This phenomenon does not occur in aqueous solution, and is most evident with the para-isomeride and least with the meta. It has also been observed in the case of tetramethyldiaminobenzophenone and the aminobenzaldoximes. The phenomenon is apparently due to an increased activity of the amino-group, and hence affords an explanation of the catalytic action of mineral acid on the acetylation of amino-compounds.

DISCUSSION.

Dr. HEWITT inquired if the absorption spectrum of *p*-aminobenzaldehyde in presence of a small quantity of hydrochloric acid was measured in absolute alcoholic solution, and he also wished to know how the substance was isolated in a pure condition.

Mr. BALY, in reply to Dr. Lowry, said that the original compound was restored when the acid was neutralised. In reply to Dr. Forster, the absorption spectrum of the hydrochloride was entirely different from that of the base in presence of a trace of alcoholic hydrogen chloride. Considerable difficulty had been experienced in the preparation of *p*-aminobenzaldehyde. It was obtained from *p*-aminobenzaldoxime and then regenerated from its hydrochloride. The melting-point was correct, and, moreover, the condensation product was very different from the aminobenzaldehyde itself in that it was only very slightly soluble in alcohol.

*241. "*Organic Derivatives of Silicon.* Part VIII. *The Resolution of* dl-*Sulphobenzylethylisobutylsilicyl Oxide and the Properties of the Optically Active Acids.*" By BERNARD DUNSTAN WILKINSON LUFF and FREDERIC STANLEY KIPPING.

dl-*Sulphobenzylethylisobutylsilicyl oxide*,—

$$[SO_3H\cdot C_6H_4\cdot CH_2\cdot SiEt(C_4H_9)]_2O,$$

is resolved into its optically active components by the fractional crystallisation of its salt with *d*-methylhydrindamine; the pure *dAd*B-salt obtained melts at 207—209°, and has $[\alpha]_D$ + 16·7° in methyl-alcoholic solution. The specific rotation of the sodium salt of the *d*-acid is $[\alpha]_D$ + 10·4°. The pure *lAl*B-salt (melting at 207—209° and having $[\alpha]_D$ 17·0° in methyl-alcoholic solution) is obtained by fractionally crystallising the salt of the *dl*-acid with *l*-methylhydrindamine; the specific rotation of the sodium salt of the pure *l*-acid is $[\alpha]_D$ - 10·5°.

d-*Methylhydrindamine* l-*sulphobenzylethylisobutylsilicyl oxide* and the corresponding *dAl*B-salt, prepared by combining the optically pure acids with *d*- and *l*-methylhydrindamine respectively, are oils at the ordinary temperature.

l-*Menthylamine* d-*sulphobenzylethylisobutylsilicyl oxide* crystallises well (with $4H_2O$), melts when anhydrous at 254—258°, and its specific rotation in methyl-alcoholic solution is $[\alpha]_D$ - 13·9°. The salt of the *l*-acid is very similar in properties, but melts at 253—257°, and has $[\alpha]_D$ 17·2° in methyl-alcoholic solution. The salt of the *dl*-acid melts at 240—245°, and has $[\alpha]_D$ - 14·7°.

d-*Bornylamine* d-*sulphobenzylethylisobutylsilicyl oxide* melts at 209—211°; the salt of the *l*-acid melts at 210—212°.

d-*Cinchonidine* d-*sulphobenzylethylisobutylsilicyl oxide* crystallises in needles melting at 171—173°, whereas the salt of the *l*-acid melts at 181—183°; the specific rotations of the two salts, however, are practically identical, having the values $[\alpha]_D$ - 68·5 and $[\alpha]_D$ - 69·3° respectively in methyl-alcoholic solution. The *cinchonidine hydrogen* salts of the *d*- and *l*-acids are crystalline, and decompose at 232—234° and 229—231° respectively.

*242. "*The Action of Phosphorus Pentachloride on the Methylene Ethers of Catechol Derivatives.* Part III. *The Cyclic Carbonates of Dichloro-ethyl- and -propyl-catechol.*" By GEORGE BARGER.

From a-3 : 4-*methylenedioxyphenyl-αβ-dichloroethane*,—

$$CH_2<^{O}_{O}>C_6H_3\cdot CHCl\cdot CH_2Cl,$$

b. p. 159—160°/12 mm., or from the corresponding *chlorohydrin*,—

$$CH_2<^{O}_{O}>C_6H_3\cdot CH(OH)\cdot CH_2Cl,$$

m. p. 95°, b. p. 169—170°/7 mm., the very unstable crystalline *substance*,—

$$CCl_2<^{O}_{O}>C_6H_3\cdot CHCl\cdot CH_2Cl,$$

m. p. 56°, b. p. 170—174°/6 mm., was obtained; the latter is transformed by formic acid into the cyclic *carbonate*,—

$$O{:}C<^{O}_{O}>C_6H_3\cdot CHCl\cdot CH_2Cl,$$

b. p. 180—182°/8 mm. Similarly, from *iso*safrole dichloride, the *substances*,—

$$CCl_2<^{O}_{O}>C_6H_3\cdot CHCl\cdot CHCl\cdot CH_3,$$

b. p. 174·5—175·5°/8 mm., and—

$$O{:}C<^{O}_{O}>C_6H_3\cdot CHCl\cdot CHCl\cdot CH_3,$$

b. p. 186—189°/8 mm., were obtained. The cyclic carbonates are readily hydrolysed to catechol derivatives, but the latter are so unstable that they have not as yet been obtainable in the pure state.

*243. "*The Synthesis of Thionaphthen Derivatives from Styrenes and Thionyl Chloride.*" By GEORGE BARGER and ARTHUR JAMES EWINS.

When styrene halides or allied derivatives are heated with a large excess of thionyl chloride to 180—270°, sulphur compounds result, which are most probably derivatives of thionaphthen. More or less extensive chlorination always takes place; thus *αβ*-dibromostyrene, $C_6H_5\cdot CHBr\cdot CH_2Br$, yields *hexachlorothionaphthen*, C_8Cl_6S, m. p. 158°. A number of similar derivatives from other styrenes were described.

244. "*The Affinity Constants of Bases as determined by the Aid of Methyl-orange.*" By VICTOR HERBERT VELEY.

It was shown that the methyl-orange method, as described in a previous communication (*Trans.*, 1908, xciii., 652 *et seq.*), gives results which are concordant with those obtained by the catalysis of methyl-acetate, inversion of sucrose, and electric conductivity methods. If the last named give accurate results, then the same applies equally to the first, which possesses the advantages of simplicity of apparatus, brevity, and smaller amount of material required.

Determinations have been made of the hydrolysis and affinity constant values of about 50 cyclic bases derived from benzene, pyridine, naphthalene, and quinoline; the mutual relationship and effects produced by the substitution of one or more atomic proportions of hydrogen, whether in the nucleus or side-chain, by various elements or groupings were discussed.

The variation of the several constants, namely, $C_{(u)}$, K_w, and K_b, with temperature was dealt with, and it was shown that in cases which determinations of these constants have been made by the same or different trustworthy methods, the experimental results obtained are in accordance with those calculated by the formula $K_{T1}/K_T = (T_1/T)^m$ (T = absolute temperature). Thus ionic dissociation, whether of water or electrolytes, cannot be differentiated in respect of temperature from any other change, generally regarded, as of a purely chemical nature. The importance of this formula for the purpose of other calculations was also emphasised.

245. "*Study of the Constitution and Properties of the Rhodanides of Inorganic Radicles.*" By AUGUSTUS EDWARD DIXON and JOHN TAYLOR.

The mean refraction value of the ·NCS group in organic thiocarbimides being about 28·1, as calculated from the formula—

$$M_D = \frac{(\mu_D - 1)p}{dt°/4°},$$

whilst that for the ·SCN group in various organic thiocyanates is about 23·3, the authors, by measuring the molecular refraction of the rhodanides, $P(CNS)_3$ and $PO(CNS)_3$, have endeavoured to ascertain the refraction effect of the rhodanic groups in these substances, with the object of deciding whether the groups have the configuration ·NCS or ·SCN.

The number found for the rhodanic group (assuming the three to be of equal value) being in each case near to 28, the authors conclude that, so far as the results of this purely physical method may be relied on as affording trustworthy evidence with respect to the atomic linking, the two rhodanides mentioned have the thiocarbamidic constitutions $P(\cdot NCS)_3$ and $PO(\cdot NCS)_3$ respectively.

Phosphoryl trithiocarbimide yields definite additive compounds with one and with two molecular proportions of aniline, or with one of diphenylamine, the products being nearly insoluble in cold water. By prolonged contact with cold water, by treatment with hot water, or by dissolution in aqueous alcohol, they are hydrolysed, yielding one-third of the contained sulphur as substituted thiocarbamide, and two-thirds as free or combined thiocyanic acid. Phosphorus trithiocarbimide (1 mol.), like the phosphoryl analogue, is precipitated completely from benzene solution by one molecular proportion of aniline; the solid product, when dissolved in cold alcohol, gives the reactions of a thiocarbamide, and is hydrolysed readily into one molecule of phenylthiocarbamide and two molecules of thiocyanic acid. The authors suggest that very pronounced electropositive or electro-negative character in the inorganic radicle may have power to influence the configuration of the rhodanic group with which it is associated.

246. "*The Electrical Conductivity of Phosphoric Acid.*" By HARRY EDWARD WILLIAM PHILLIPS.

Pure phosphoric acid was prepared by treating re-distilled phosphorus trichloride with specially purified water at an equable temperature, and oxidising the resulting phosphorous acid by means of pure nitric acid. The hydrochloric acid and the excess of nitric acid were then expelled by heat.

The phosphoric acid thus prepard was a transparent colourless oily liquid, which showed no tendency to crystallise in the higher strengths, but a specimen of 43 per cent solution yielded numerous small, soft, gelatinous crystals spontaneously on cooling. The pure acid neither reddened litmus nor decomposed carbonates.

For the measurement of conductivity, Kohlrausch's telephone method was employed, and a special Wheatstone bridge was made by the author for the purpose, which gave opportunity of very accurate work.

Curves were drawn showing the actual conductivity per cent of the acid at 0°, also a set of curves showing the comparative conductivity per cent of sulphuric, nitric, hydrochloric, and phosphoric acids and sodium and potassium hydroxides. The results obtained were also worked out to molecular conductivities, and these were compared with the nine values previously obtained by Kohlrausch at 18°, and the more extended work of Jones and Bassett at 0°; they were also compared with the corresponding values for the three common mineral acids. Finally, curves of dissociation values, obtained by means of the formula $\frac{\Lambda}{\Lambda_\infty}$, were drawn.

The results seem to bear out the theory of indicators regarding the ionisation of the hydrogen atom in the molecule of phosphoric acid.

247. "*The Molecular Volumes of the Nitrites of Barium, Strontium, and Calcium.*" By PRAFULLA CHANDRA RAY.

The molecular volumes of the nitrites of barium, strontium, and calcium in their monohydrated forms are 77·87, 74·59, and 67·24, whilst those of the anhydrous salts are 70·90, 62·54, and 57·56 respectively. The radicle NO_2 of the anhydrous nitrites of these metals occupies a constant volume, namely, 24·2.

248. "*The Formation of some Carbides.*" By JOHN NORMAN PRING.

An investigation has been made of the direct union of carbon with the elements aluminium, silicon, and iron in the absence of solid and gaseous impurities. Some preliminary experiments were also carried out with magnesium.

An intimate mixture of the metal with highly-purified carbon, both in a very fine state of division, was heated in a suitable apparatus, enabling definite constant temperatures to be maintained over long intervals, and capable of being evacuated to a pressure below 0·01 mm.

The direct union of pure silicon and carbon in a vacuum commences between 1250° and 1300°, and the presence of iron does not apparently facilitate the reaction, nor does carbon monoxide exert any influence between the limits of atmospheric pressure and 0·03 mm.

Aluminium unites directly with pure carbon in a vacuum at its melting-point (650°), forming aluminium carbide, Al_4C_3, and the velocity of the reaction increases with the temperature, proceeding rapidly above 1400°.

Iron unites directly with carbon at about 700° under a pressure of about 0·05 mm.

Magnesium shows evidence of forming a new carbide below 600°, this compound giving some saturated hydrocarbon on decomposition with water or acids.

249. "*4-Chloroacenaphthene.*" By HOLLAND CROMPTON and EVA RICHARDIS CYRIAX.

When acenaphthene in chloroform solution is treated with chlorine (1 mol.) and the solvent evaporated, an oil remains which does not crystallise on standing and from which no crystalline product could be obtained by means of solvents. On distillation, it yields some unchanged acenaphthene, and then, as main product, a distillate passing over at 305—310° which solidifies in the receiver. This consists of almost pure 4-chloroacenaphthene.

4-*Chloroacenaphthene* crystallises from alcohol in colourless leaflets; it melts at 65°, and boils at 309°; its *picrate* crystallises in yellow needles, melting at 133°. On oxidation by sodium dichromate in acetic acid solution, it yields a chloronaphthalic acid, the anhydride of which melts at 198° and the imide at 278°. When the calcium salt of this acid is heated with soda-lime, naphthalene is obtained, mixed with a little α-chloronaphthalene. If the copper salt is used in place of the calcium salt, α-chloronaphthalene is produced with little naphthalene. On fusing with potassium hydroxide at 250—260°, the acid is converted into 4-hydroxynaphthalic acid, the anhydride of which melts at 257°; this acid had previously been obtained by Graebe from 4-bromonaphthalic acid (*Annalen*, 1903, cccxxvii., 77). The formation of this 4-hydroxynaphthalic acid and of α-chloronaphthalene show that the acid is 4-chloronaphthalic acid, and confirm the constitution given to the chloroacenaphthene.

250. "*Some Molecular Compounds of Styphnic and Picric Acids.*" By CHARLES STANLEY GIBSON.

The following compounds have been prepared :—

Acenaphthene styphnate, $C_{12}H_{10},C_6H(OH)_2(NO_2)_3$, crystallising in long orange-coloured prisms melting at 153—154° ; *phenanthrene styphnate*, $C_{14}H_{10},C_6H(OH)_2(NO_2)_3$, crystallising in yellowish red needles melting at 125—126° ; *bromonaphthalene styphnate*, $C_{10}H_7Br,C_6H(OH)_2(NO_2)_3$, crystallising in small, bright yellow needles melting at 107 — 108° ; m-*nitroaniline styphnate*, $NH_2{\cdot}C_6H_4(NO_2),C_6H(OH)_2(NO_2)_3$, crystallising in pale yellow needles melting at 137° ; and m-*nitroaniline picrate*, $NH_2{\cdot}C_6H_4(NO_2),C_6H_2(OH)(NO_2)_3$, crystallising in yellow radiating needles melting at 143°.

NOTICES OF BOOKS.

The Air and Ventilation of Subways. By GEORGE A. SOPER, Ph.D. New York: John Wiley and Sons. London : Chapman and Hall, Limited. 1908.

IN this book the reports which have appeared in the "Transactions of the Rapid Transit Commissioners" are summarised, and edited in such a way as to make them interesting to the general public. Additional matter relating to the characteristics of good and bad air and the general theories of ventilation has been worked up to form a preface for the benefit of those who have had no training in sanitary science ; in the chapter on methods of ventilating, the formulæ for calculating the velocity of air, quantities of air delivered, &c., are collected, but it has not been considered necessary to explain in full the methods of arriving at the formulæ, to follow which would require some considerable knowledge of the principles of mechanics. The systems of ventilation in use in the subways of various large towns are described, and special attention must be called to the diagrams showing the air currents set up by express and other trains ; in the preparation of these much ingenuity has been expended, and the results are most interesting. The bacteriological and chemical methods of analysing the air are illustrated graphically by many charts ; the investigations related to temperature, humidity, odour, bacteria, dust, and carbon dioxide. Oxygen determinations were discontinued after about eighty had been made, for the differences obtained for subway and street air were only trifling. Ozone was not determined, and the author appears to favour the opinion that it does not actually exist in the air. The final chapter deals with the health of subway employés, which has been subjected to a careful and thorough investigation ; though possibly some of the figures tabulated are slightly misleading, on the whole the results obtained are not disquieting ; the presence of the black metallic dust, composed principally of iron, appears to constitute the most serious source of danger to health.

Thirty-second Annual Report of His Majesty's Inspectors of Explosives. London : Wyman and Sons, Ltd. 1908.

THE annual report of His Majesty's Inspectors of Explosives for the year 1907 contains the usual tables relating to accidents, authorised explosives, &c. In the report of the chemical advisers details are given of the recently worked out methods of testing various kinds of explosives for mercury. It has been found that specially constructed spectrum tubes are necessary, and different explosives require various modifications of the method, which is very delicate and reliable. A report is also included on some experiments on the dangers of coal-dust, samples of which were subjected to tests under varying conditions, to determine under what circumstances they were dangerous, and whether any relations could be established between the constituents of a coal-dust and its sensitiveness.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 17, October 26, 1908.

Mercurous Nitrate as a Micro-chemical Reagent for Arsenic.—G. Denigès.—The author has recently shown that characteristic crystals of arsenic, as arsenic acid, can be obtained on a glass plate by the use of ammoniacal or acetic silver nitrate or by magnesia mixture. By very slightly modifying the method, mercurous nitrate can be used for the same purpose, and similar crystals are obtained provided that the detailed directions given by the author are carefully followed.

Oxydasic Phenomena produced by Colloidal Iron Ferrocyanide.—J. Wolff.—In a feebly alkaline solution colloidal iron ferrocyanide acts like an oxydase towards hydroquinone, and the phenomenon in continuity and the disparity between cause and effect may be compared with the most energetic diastatic actions. It may be explained by supposing that the alkali favours the oxidation of the phenol, and consequently its attack by the iron salt acting like an oxydase. Some salts of manganese oxidise hydroquinone, and it is only necessary to add to them one hundred times less iron (in the form of ferrocyanide) than the amount of manganese present to double the velocity of the reaction.

Action of Bromine on Ether. Monobromaldehyde.—Ch. Mauguin.—A mixture of bromine, water, and ether when exposed to light is decolorised, the action being very rapid in sunlight. When all action is over, two layers of liquid are obtained ; the lower is aqueous and contains large quantities of hydrobromic acid, and the upper contains an aldehyde which gives a white crystalline solid melting at 146° with urethane. This solid is the condensation product of monobromaldehyde with urethane, and with 10 per cent sulphuric acid it yields monobromaldehyde in aqueous solution.

Berichte der Deutschen Chemischen Gesellschaft. Vol. xli., No. 14, 1908.

Preparation of Sulphinic Acids.—Emil Knoevenagel and James Kenner.—Friedel and Craft's reaction can conveniently be used to prepare aromatic sulphinic acids, and yields of on an average 80 per cent can be obtained if the reaction occurs at a low temperature and if it is started by leading in dry hydrochloric acid. The aluminium chloride formed must not be decomposed by acids but by alkalis, the alkaline salts being subsequently decomposed by acids. Phenol ethers react with sulphur dioxide and aluminium chloride without the addition of hydrochloric acid in the cold, the equation being $RO.C_6H_5 + SO_2 = RO.C_6H_4.SO_2H$. The reaction readily goes further to the sulphoxides $2RO.C_6H_5 + SO_2 = H_2O + (RO.C_6H_4)_2SO$, and thence to the sulphonium compounds—

$$3RO.C_6H_5 + SO_2 + AlCl_3 = AlCl_2.OH + (RO.C_6H_4)_3SCl.$$

Hydrochloric acid accelerates the formation of sulphinic acids, especially from aromatic hydrocarbons and sulphur dioxide in presence of aluminium chloride, so possibly the acid chloride of sulphurous acid reacts as follows :— $C_6H_6 + HSO_2Cl = HCl + C_6H_5.SO_2H$, but it is more probable that a compound of sulphur dioxide and aluminium chloride of the composition $Cl.SO_2.AlCl_2$ is formed. $C_6H_6 + Cl.SO_2.AlCl_2 = HCl + C_6H_5.SO_2.AlCl_2$.

Cerium Sulphides.—Wilhelm Biltz.—Cerium sesquisulphide can readily be obtained by sulphating cerium dioxide with sulphuretted hydrogen at a bright red heat, or by heating cerous sulphate in a current of sulphuretted hydrogen. At a lower temperature the disulphide is

formed. The sesquisulphide is a crystalline dark brown powder which is stable in air and in water. It dissolves in dilute acetic acid in the cold, and also in dilute hydrochloric and sulphuric acids. The disulphide does not appear to be a derivative of tetravalent cerium corresponding to the dioxide, but it is rather to be regarded as a polysulphide of formula $Ce_2S_3.S$. Cerium disulphide decomposes at 720°. The sesquisulphide melts between 1400° and 1500°, undergoing decomposition. It is stable in an atmosphere of pure hydrogen at a red heat.

Action of Arsenite and Cyanide on Thiosulphonic Acid Salts.—A. Gutmann.—When an arsenite or cyanide acts on thiosulphate, sulphite and monosulphoxy arsenate or rhodanate are formed :—

$$S_2O_3Na_2 + AsO_3Na_3 = SO_3Na_2 + SAsO_3Na_3,$$
$$S_2O_3Na_2 + NCK = SO_3Na_2 + NCSK.$$

The action of arsenite and cyanide on arylthiosulphonic acid salts is similar, sulphinic acids being formed instead of sulphites :—

$$RS_2O_2Me + AsO_3Na_3 = RSO_2Me + SAsO_3Na_3,$$
$$RS_2O_2Me + NCK = RSO_2Me + NCSK.$$

The liberation of sulphur by thiosulphonic acid salts depends on the formation of a yellow intermediate product, MeOSH, an alkalihydropersulphide. These reactions show the peracid character of thiosulphonic acid salts, the constitution of which must be expressed by the formula $R—S(=O)—S—OMe$.

Reaction between Silver Sulphide and Silver Sulphate. Preparation of Dark coloured Glass.—O. Sackur.—When a mixture of silver sulphide and sulphate is heated above 300°, sulphur dioxide and metallic silver are formed. Attempts to determine the equilibrium pressure of the mixture failed, the only result being the proof that the pressure is greater than ten atmospheres at 300°. If the mixture is heated in a tube of ordinary Thuringian glass the latter acquires a brown colour below 400° ; this colour becomes darker and turns reddish when the mass is left in contact with the glass for some time. It is not distributed uniformly throughout the whole mass, but only over a relatively thin surface layer. Glass coloured by this method absorbs the blue and blue-green rays of the spectrum almost completely, and the yellowish green rather less, and vessels thus coloured may very conveniently be used for photographic purposes to absorb the most actinically active rays. When the brown glass is heated in a Bunsen flame tempering colours are produced owing to the formation of a very thin layer of metallic silver on the surface. A distinct silver white layer is obtained by heating in the blowpipe flame. The brown and reddish brown glass possesses a small but decided conductivity for the electric current at the ordinary temperature.

Silicon Acids and their Derivatives.—Wilhelm Melzer.—When a molecule of magnesium halogen alkyl acts on silicon tetrachloride, silicon alkyl trichlorides are formed :—$RMgX + SiCl_4 = RSiCl_3 + MgXCl$ (where R denotes an alkyl and X a halogen). These are converted into the corresponding silicon acids of the type R.SiOOH by water, and into orthosilicon acid esters of the type $R.Si(OC_2H_5)_3$ by alcohol. Every trace of moisture must be avoided, especially in experiments with chlorides. By this method the author has prepared various alkyl silicon haloids, *e.g.*, silicon propyltrichloride, $C_3H_7.SiCl_3$, a clear liquid fuming in air and possessing a piercing smell, and also silicon acids, *e.g.*, silicobutyric acid, $C_3H_7.SiOOH$, and their esters.

Lowest Oxides of Sulphuretted Hydrogen.—Emil Fromm.—The lowest oxides of sulphuretted hydrogen are sulphur hydrate, H_2SO, and sulphoxylic acid, H_2SO_2. Sulphur hydrate is not known either free or in inorganic compounds. Derivatives of it are the sulphoxides, R.SO.R, and the hypothetical compounds, R.SOH, the existence of which has, however, never been demonstrated with certainty. All organic disulphides with adjacent double bonds of general formula $X:C.S.S.C:Y$ ($\dot{R}$ $\dot{R}_1$) are decomposed by water, alkalis, and amines, giving elementary sulphur. The author's experiments have proved that sodium tetrathionate also yields elementary sulphur, and thus decomposes in exactly the same way as organic disulphides with adjacent double bonds, thus: $Na_2S_4O_6 + H_2O = S + NaHSO_4 + NaHS_2O_3$. Sodium ethyl thiosulphate and the aromatic disulphides give no elementary sulphur on hydrolysis, but in all probability the hypothetical oxidation product R.SOH is formed. This substance is a strong oxidising agent, and is unstable in alkaline solution, readily giving mercaptan and sulphinic acid, $2R.SOH = R.SH + R.SOOH$. Sulphoxylic acid has not yet been obtained in the free state ; complicated salts of it are the hydrosulphites. A simple salt of sulphoxylic acid, zinc sulphoxylate, $ZnSO_2$, has been prepared by the action of zinc dust on an ethereal solution of SO_2Cl_2, but the compound has not yet been isolated. Aldehydes react with hydrosulphite in presence of alkali according to the equation—

$$R.CHO + Na_2S_2O_4 + NaOH = R.CH(OH).SO_2Na + Na_2SO_3.$$

The acid sulphoxylate may be added to the aldehyde in such a way that oxysulphinic acid is formed, $R.CH(OH).SO_2Na$, or esters of sulphoxylic acid may result, $R.CH(OH).O.SO.Na$ or $R.CHOH.O.SNa$ (Ö), or derivatives of orthosulphoxylic acid of formula $R.CH.O.S.Na$ (O, O—OH). The investigation of the aldehyde and ketone sulphoxylates, especially rongalite (formaldehyde sulphoxylate), shows that in all probability rongalite is an ester of sulphoxylic acid and not an oxymethane sulphinic acid. Its formula may be—

$$HO.CH_2.O.SO.Na \quad \text{or} \quad H_2C(O....).O.S.Na,\ (\dot{O}H)$$

and possibly it reacts according to both formulæ. In the dibenzyl derivative the Na and the H of the hydroxyl group are both replaced by C_7H_7.

Carbon Suboxide.—Otto Diels and Leonid Lalin.—Although carbon suboxide is unsaturated, it shows only a comparatively slight tendency to form addition products except with formic and acetic acids—addition products with which are readily obtained. They have, however, been prepared in small quantities only, for even at low temperatures the two substances react according to the equation $C{\equiv}CO{\equiv}CO + 2HCO.OH = 2CO + CH_2{<}^{COOH}_{COOH}$. Liquid sulphur dioxide is a solvent in which the addition of formic acid to carbon suboxide can very conveniently be studied. The addition product is thus found to be a crystalline substance which is quite stable as long as precautions are taken against the presence of moisture and a rise of temperature. The compound contains two molecules of formic acid to one of carbon suboxide, and corresponds to the empirical formula $\left[C{\equiv}CO{\equiv}CO + 2H.COOH\right]$. It seems probable that the characteristic linkage of carbon suboxide is still present in the compound, and that it is a mixed anhydride of malonic and formic acids, although not of normal constitution. Its formula appears to be $C{<}^{C{<}^{OH}_{COOH}}_{C{<}^{COOH}_{OH}}$. On heating, the molecule is decomposed into 2CO and $CH_2{<}^{COOH}_{COOH}$. Acetic acid and carbon suboxide also react in sulphur dioxide, the product being a syrup which, when distilled in a low vacuum, gives acetic acid anhydride and leaves a syrupy residue. The

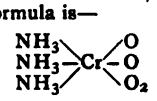
latter probably consists of monomolecular malonic acid anhydride, for with water it yields almost the theoretical quantity of malonic acid.

Formic and Acetic Acid Salts of Chromium.—A. Werner.—The formates and acetates of chromium can be divided into two groups:—(*a*) Normal salts; (*b*) complex salts. The normal salts may be prepared in crystalline form by adding the organic acid to the dihydroxysulphate—

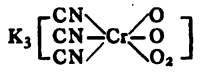
$$\left[Cr\begin{matrix}(OH)_2\\(OH_2)_4\end{matrix}\right]_2SO_4 + 6HX = 2\,[Cr(OH_2)_6]X_3 + H_2SO_4.$$

The formate is greenish grey and the acetate blue-violet. The stability decreases as the magnitude of the fatty acid residue increases, the butyrate being so unstable that it decomposes spontaneously after a few days. All fatty acid hexaquochromium salts dissolve in much water with a blue-green coloration, due to hydrolytic dissociation. When moist chromium hydrate is heated with formic acid till it all dissolves, dark green crystals separate out on cooling. These have the formula $Cr_3\begin{matrix}(OH)_2\\(O.COH)_7\end{matrix} + 5H_2O$. With nitric acid they give crystals of the composition $[Cr_3(O.COH)_6(OH)_2]NO_3 + 5H_2O$. Other salts of the same type can be prepared by the action of mineral acids on the nitrate.

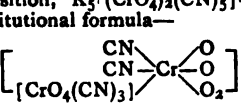
Chromium Tetroxide Compounds.—E. H. Riesenfeld.—Chromium tetroxide compounds are not perchromates but belong to a special class of substances. To determine their composition the molecular weight of chromium tetroxide triammin was ascertained, and it was found that the constitutional formula is—



When treated with potassium cyanide a tricyanide of formula—



is obtained. When an aqueous solution of chromic acid is treated with potassium cyanide and hydrogen peroxide, according to the concentration the tricyanide is formed, or a salt of composition, $K_5[(CrO_4)_2(CN)_5] + 5H_2O$. This last has the constitutional formula—



and is the first known case of a complex salt in which a complex occupies a co-ordination position in a complex salt exactly similar to itself. It seems most probable that the chromium tetroxide compounds are to be regarded as compounds of hexavalent chromium.

Oxalyl Chloride.—H. Staudinger.—A good yield of oxalyl chloride may be obtained by allowing a molecule of anhydrous oxalic acid to act on two molecules of phosphorus pentachloride. A lower yield of the same product is obtained if hydrated oxalic acid is used. Oxalyl chloride is a colourless liquid boiling at 64°. With water it yields carbon dioxide, carbon monoxide, and hydrochloric acid quantitatively, no oxalic acid being formed. With dimethylaniline it gives dimethylaminobenzoylformic acid chloride, or with excess of the base tetramethyldiaminobenzile.

MISCELLANEOUS.

Royal College of Science.—The first Annual Dinner of old students of the Royal College of Science is to be held at the Criterion Restaurant on December 9th. Mr. H. G. Wells (1884-87), who was the first editor of the College Magazine, will preside, and the guests will include Mr. Arthur Acland, Sir Francis Mowatt, Mr. Grant Ogilvie, Dr. Bovey, Dr. Miers, and Professors Tilden and Judd. Tickets may be obtained from Mr. T. Ll. Humberstone, Hon. Secretary of the Dinner Committee, 3, Selwood Place, London, S.W. It is expected that the Dinner will lead to the formation of an Association of Old Students of the College.

Institute of Chemistry.—The following candidates passed the Final Examination for the Associateship in the Branch of Biological Chemistry, held from Oct. 19 to 23:—E. B. Anderson, B.Sc. (Lond. and Birm.); F. G. S. Baker, M.A. (Oxon.): H. L. Gammon; and A. R. Smith, B.Sc. (Manchester). The following Associate also presented himself for the Examination, and was awarded the Certificate: J. H. Tetton, B.A., B.Sc. (R.U.I.). The Examiner in Biological Chemistry was Dr. Arthur Harden, F.I.C., and the Examiners in General Chemistry were Mr. Bertram Blount, F.I.C., and Professor Herbert Jackson, F.I.C.

The Royal Society.—At the Annual Meeting, November 30th, 1908, the following Officers and Council were elected:—President: Sir Archibald Geikie, K.C.B., D.C.L., Sc.D., LL.D. Treasurer: Alfred Bray Kempe, M.A., D.C.L. Secretaries: Prof. Joseph Larmor, D.Sc., D.C.L., LL.D., and Prof. John Rose Bradford, M.D., D.Sc. Foreign Secretary: Sir William Crookes, D.Sc. Other Members of the Council: Sir George Howard Darwin, K.C.B.; Prof. James Cossar Ewart, M.D.; Sir David Gill, K.C.B.; John Scott Haldane, M.D.; Charles Thomas Heycock, M.A.; Prof. Horace Lamb, D.Sc.; Prof. Hector Munro Macdonald, M.A.; Frederick Walker Mott, M.D.; The Hon. Charles Algernon Parsons, C.B.; Prof. William Henry Perkin, Ph.D.; Prof Edward Bagnall Poulton, D.Sc.; Lieut.-Col. David Prain, C.I.E.; Sir Arthur William Rücker, D.Sc.; Rt. Hon. Sir James Stirling, M.A.; Prof. Frederick Thomas Trouton, Sc.D.; William Whitaker, B.A.

Physical Society Exhibition.—At the Exhibition to be held on Friday evening, December 11 (from 7 to 10), at the Royal College of Science, South Kensington, the following firms will be exhibiting:—Messrs. Cambridge Scientific Instrument Co.; Casella and Co.; A. C. Cossor, Ltd.; J. H. Dallmeyer, Ltd.; Elliott Bros.; Everett, Edgcumbe, and Co.; Gallenkamp and Co., Ltd.: Gambrell Bros.; J. J. Griffin and Sons, Ltd.; A. Hilger, Ltd.; Indiarubber, Gutta-percha, and Telegraph Works Co.; Marconi's Wireless Telegraph Co.; Nalder Bros. and Thompson; Newton and Co.; R. W. Paul; James Pitkin and Co.; Siemens Bros. and Co.; Snell and Tinsley; J. Swift and Son; Alexander Wright and Co.; and Carl Zeiss, Ltd. From the Programme, of which we have received an advance proof, there appear to be many items of considerable interest to both physicists and electrical engineers. We understand that invitations have been given to the Institution of Electrical Engineers, the Faraday Society, the Optical Society, and the Röntgen Society. Admission, however, except to Fellows of the Physical Society, will be by ticket only, and therefore members of the Societies just mentioned desiring to attend the Exhibition should apply to the Secretary of the Society to which they belong.

MEETINGS FOR THE WEEK.

MONDAY, 7th.—Royal Society of Arts, 8. (Cantor Lecture). "Twenty Years Progress in Explosives," by O. Guttmann.
—— Society of Chemical Industry, 8. "Cellulose and Chemical Industry," by C. F. Cross and E. J. Bevan.

WEDNESDAY, 9th. Royal Society of Arts, 8. "Kinematography in Natural Colours," by G. A. Smith and C. Urban.

THURSDAY, 10th.—Royal Society of Arts, 4.30. "The Birds of India," by Douglas Dewar, I.C.S.

FRIDAY, 11th.—Physical, 7. (At the Royal College of Science, South Kensington). Exhibition of Apparatus.

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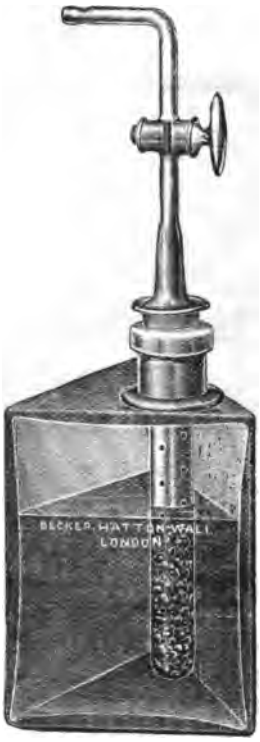

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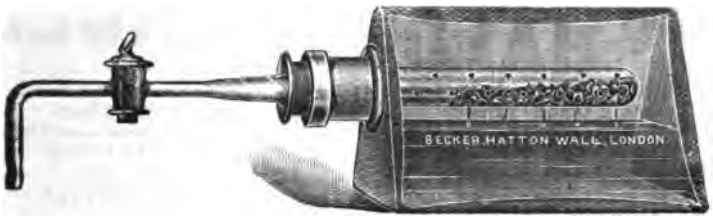

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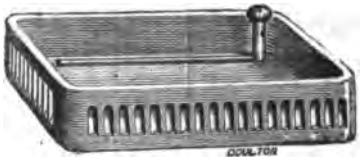

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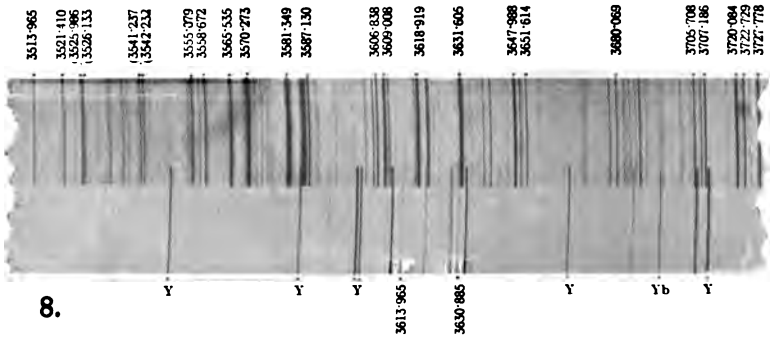
THE SCANDIUM SPECTRUM.

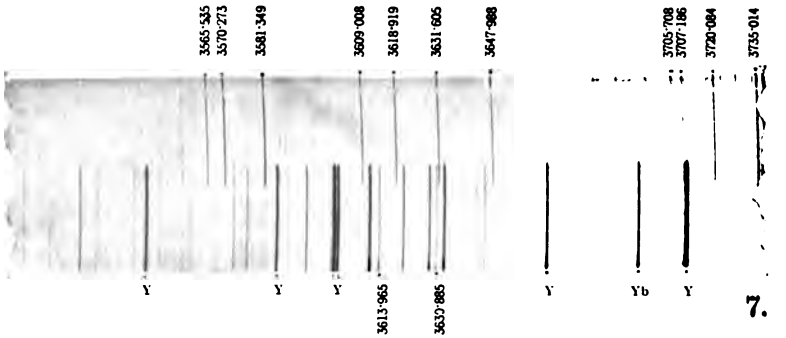
SELECTED GROUPS OF CHARACTERISTIC LINES.

To economise space and avoid unnecessary complications, I give photographs of only those parts of the spectrum containing the most prominent scandium groups. The upper half of each strip shows the iron lines used as standards, with their wave-lengths according to ROWLAND'S measurements. The lower halves contain the scandium lines, with their wave-lengths calculated from the iron standards.

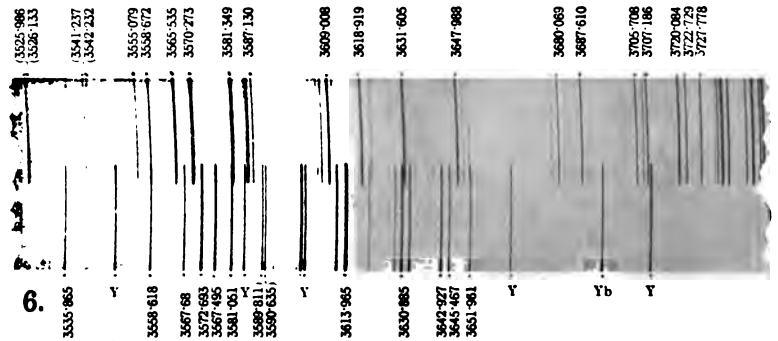
There are also many strong lines in the ultra-violet spectrum of scandium between λ 3535·865 and λ 2300. I have not completed the measurements of these lines, and their description must form the subject of a subsequent communication.



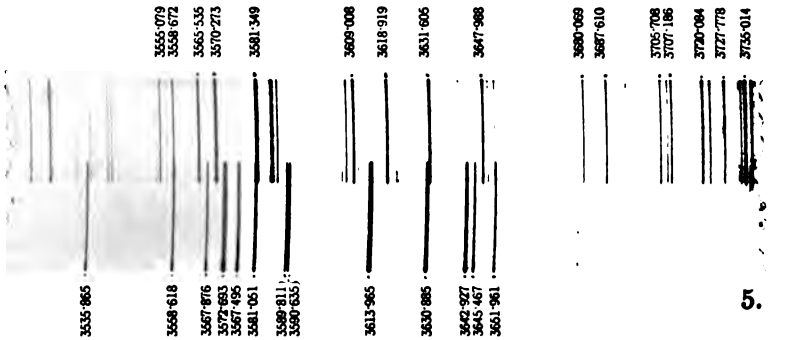
8. Spectrum of crude rare earths from keilhauite, also photographed in the same position as in Spectrum 5. It was from the rare earths from keilhauite that CLEVE prepared scandium.



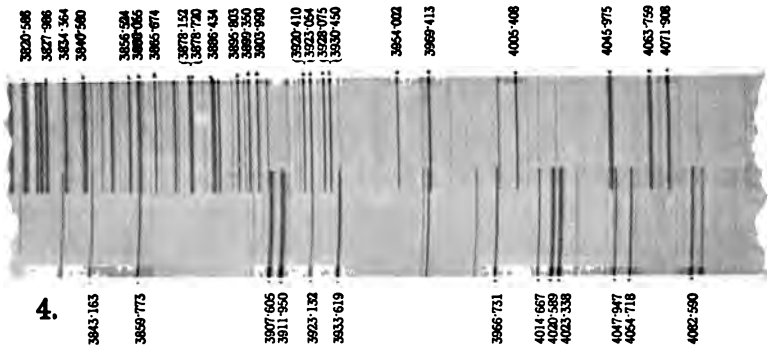
7. Spectrum of crude rare earths from euxenite, also photographed in the same position as in Spectrum 5. It was from euxenite that NILSON first extracted scandium.



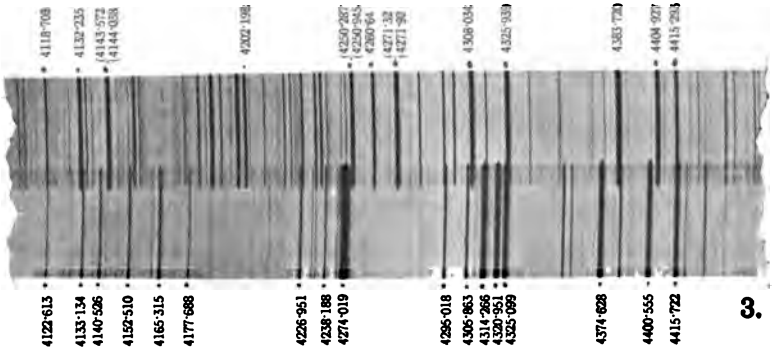
6. Spectrum of crude rare earths from wiikite, photographed in the same position as in Spectrum 5. In addition to the dominant and other lines of scandium, which are marked in wave-lengths, there are seen lines of yttrium and one strong line of ytterbium.



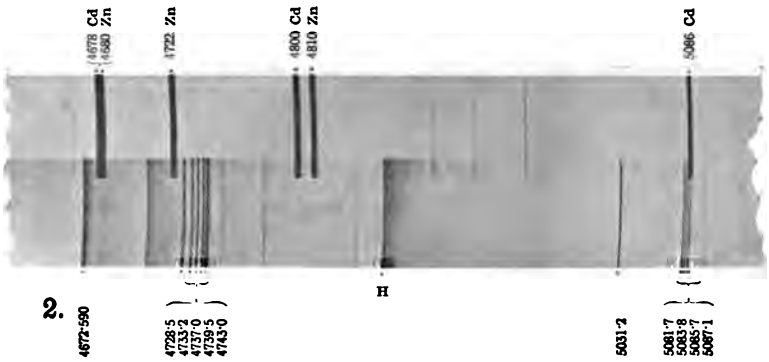
5. Group of lines between λ 3651·961 and λ 3535·865. This is the part of the spectrum which I photograph when minerals are being examined for scandium. The "dominant line," λ 3613·965 occurs here between the iron lines λ 3618·919 and λ 3609·008.



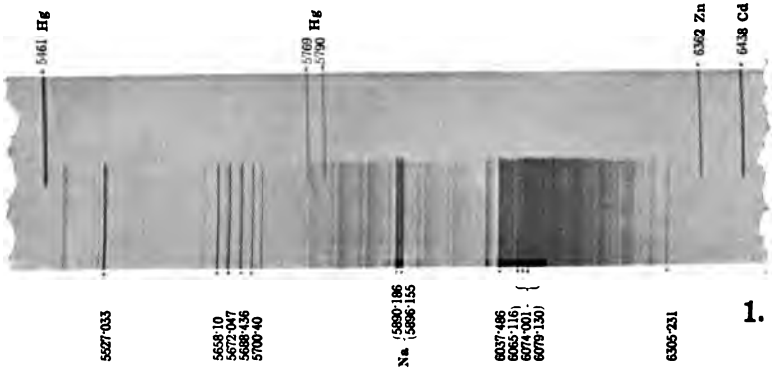
4. Group of lines between λ 4082·590 and λ 3843·163.



3. Group of lines between λ 4415·722 and λ 4122·613.



2. Group of lines between λ 5087·1 and λ 4672·590.



1. Group of visible lines between λ 6305·231 and λ 5527·033.

In the part of the spectrum shown on strips 1 and 2 there are no iron lines suitable for standards. Here, therefore, I have used some good lines of zinc, cadmium, and mercury. Attention is drawn to the comparative intensities of the yttrium lines and the dominant line of scandium in the spectra from the wiikite, euxenite, and keilhauite earths. The spectrum of the earths from gadolinite is not given, as the dominant line of scandium in it is too faint to be reproduced.

THE

CHEMICAL NEWS

AND

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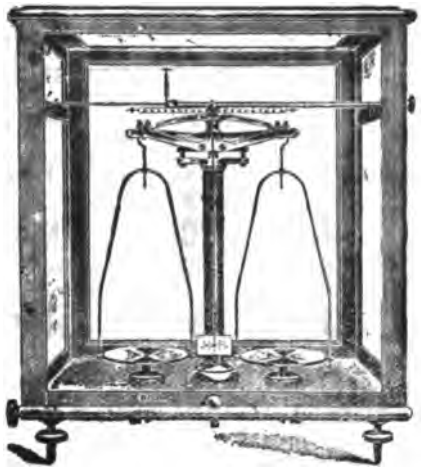

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THE CHEMICAL NEWS.

Vol. XCVIII., No. 2559.

ON SCANDIUM.*

By Sir WILLIAM CROOKES, D.Sc., F.R.S.

(Continued from p. 277).

Scandium Sulphate, $Sc_2(SO_4)_3,6H_2O$.

Scandium hydroxide easily dissolves in dilute sulphuric acid; after ignition the oxide dissolves with difficulty in dilute, but readily in strong acid, with evolution of heat. The excess of sulphuric acid must be driven off carefully, when the anhydrous sulphate is left behind in the form of a fine white powder.

Nilson gives the formula of the anhydrous salt, $Sc_2O_6,3SO_2$. He says: "It loses acid at a high temperature, and leaves pure scandia. The salt treated with water only gives at first a milky liquid, the anhydrous salt combines very slowly with water, and then re-dissolves. On heating, a clear solution is immediately obtained." According to Cleve, "Sulphuric acid gives with scandia a white and bulky mass of sulphate, resembling thorium sulphate when it separates by heat. The sulphate does not form distinct crystals." It is very soluble in water, and a strong solution when heated does not deposit crystals, in that respect differing from most of the other rare-earth sulphates. The solution may be evaporated to a supersaturated syrup, which, standing in the cold for some hours, deposits crystals of the 6-hydrate. One hundred parts of the saturated solution at 12° were found to contain 44·5 parts of anhydrous scandium sulphate. Scandium sulphate is not deliquescent in the ordinary air of a room even in damp weather, and is insoluble in alcohol. On mixing 100 grs. of anhydrous scandium sulphate with the same quantity of water, the temperature rose in a few minutes from 14° to 21°.

On adding absolute alcohol to a stroug solution of scandium sulphate the solution becomes milky, and a heavy oily-looking liquid sinks to the bottom. This may be separated by filtration through paper moistened with alcohol, when the residue is left as a viscous liquid which on exposure to the air becomes thicker. This oily-looking liquid has the composition of the 6-hydrate sulphate.

When strongly heated, the sulphate decomposes, leaving pure scandia. It is, however, difficult to drive off all the sulphuric acid from the sulphate even by heating the crucible containing it before the blowpipe. The best way to decompose scandium sulphate is first to ignite it to the highest temperature of a Bunsen burner, then to moisten with water and add a little ammonium carbonate. Dry and ignite again, and pure scandia is left free from sulphuric acid.

Nilson says: "This salt separates from a syrupy solution in small globular aggregates. It is inalterable in the air, it loses four molecules of water at 100°, and the rest at a higher temperature." He found on analysis 14·26, 14·46, and 7·16, 7·29 of H_2O, instead of 14·88 and 7·44.

I have crystallised this sulphate from a syrupy aqueous solution; it is dried by repeated pressure between many folds of blotting-paper.

Analysis.

1. 18·156 grs. of the crystallised 6-hydrate, finely powdered, and heated to 100° for three hours, lost 2·602 grs. of water, leaving a 2-hydrated sulphate.
2. 20·725 grs., kept in a desiccator over sulphuric acid for 170 hours, lost 1·561 grs. of water, leaving a 4-hydrated sulphate.
3. 29·138 grs. left on ignition 8·068 grs. of scandia.
4. 31·508 grs. of the liquid sulphate, precipitated from solution on adding alcohol containing a little ether, were dissolved in water, and the scandia precipitated by ammonia. When dried and ignited it weighed 8·624 grs.

	Theory.		Experiment.
			1, 2, 3, and 4.
Sc_2O_3	136·200	28·11	27·53
$3SO_3$	240·180	49·58	50·61
$2H_2O$	36·032	7·44	7·53
$4H_2O$	72·064	14·87	14·33
	484·476	100·00	100·00

The 6-hydrate effloresces in a dry atmosphere, and loses one molecule of water, becoming the 5-hydrate, which appears to be the most stable hydrate at ordinary temperatures:—

$Sc_2(SO_4)_3,5H_2O$.

Analysis.

1. 16·619 grs. of the 5-hydrate, heated to 150°, lost 3·206 grs. of water.
2. 16·619 grs., after drying as described and igniting to a yellow heat, left 4·878 grs. of scandia.
3. 15·608 grs. of the 5-hydrate, heated *in vacuo* at 100° for twenty hours, and then for ninety minutes at 150°, lost 3·153 grs. of water. Ignited to a full yellow heat, it left 4·558 grs. of scandia.
4. 38·819 grs., heated to just below visible redness, lost 8·019 grs. of water. (At this temperature a trace of basic sulphate is formed. This accounts for the excess over the theoretical loss of water).

	Theory.		Experiment.				Mean.
			1.	2.	3.	4.	
Sc_2O_3 ..	136·20	29·20	—	29·35	29·20	—	29·27
$3SO_3$ ..	240·18	51·49	—	—	—	—	50·68
$5H_2O$ ..	90·08	19·31	19·29	—	20·20	20·66	20·05
	466·46	100·00					100·00

Anhydrous Scandium Sulphate.

$Sc_2(SO_4)_3$.

When the hydrated sulphate is heated to 250° it loses the whole of the water and becomes anhydrous. Its composition now is $Sc_2(SO_4)_3$.

At a bright yellow heat all the sulphuric acid goes off.

Analysis.

1. 30·716 grs. left on ignition 11·092 grs. of scandia.
2. 18·907 grs. left on ignition 6·827 grs. of scandia.

Synthesis.

3. 6·817 grs. of ignited scandia, converted into sulphate, yielded 18·907 grs. of scandium sulphate.

	Theory.		Experiment.		
			1.	2.	3.
Sc_2O_3 . ..	136·20	36·19	36·11	36·11	36·06
$3SO_3$	240·18	63·81	63·89	63·89	63·94
	376·38	100·00	100·00	100·00	100·00

When the anhydrous sulphate is heated to dull redness it loses one molecule of SO_3, and becomes a basic salt of the composition—

$Sc_2O(SO_4)_2$.

Synthesis.

4·622 grs. of ignited scandia were dissolved in strong sulphuric acid, the excess of acid driven off by heat, and the dry sulphate heated to dull redness. It weighed 10·121 grs.

* A Paper read before the Roya Society, April 30, 1908.

	Theory.		Experiment.
Sc_2O_3	136·20	45·97	45·67
$2SO_3$	160·12	54·03	54·33
	296·32	100·00	100·00

Scandium and Potassium Double Sulphate, $3K_2SO_4,Sc_2(SO_4)_3$.

The behaviour of the double sulphates of potassium and the rare-earth metals is usually considered to govern their position in the broad groups into which they are divided. If the double sulphate is insoluble in a saturated solution of potassium sulphate the earth is supposed to belong to the cerium group, while if the double sulphate is soluble the earth is said to be one of the yttrium group. According to Cleve (*Comptes Rendus*, vol. lxxxix., p. 419, August 18, 1879):—"Potassium sulphate in concentrated solution gives rise to the separation of a double salt, a crystalline powder, soluble in a saturated solution of potassium sulphate." Cleve gives the composition of the double salt as $Sc_2O_3,3SO_3+2K_2O,SO_3$. On the other hand, Nilson says (*Comptes Rendus*, vol., cxi., p. 118, July 12, 1880):—"Scandium sulphate in a saturated solution of potassium sulphate gives a double salt, completely insoluble. . . . It dissolves with difficulty in water, even boiling, and not at all in a saturated solution of potassium sulphate. . . . The composition of the double salt, $3K_2O_2SO_2+Sc_2O_63SO_2$, shows that scandium belongs to the group of gadolinite and cerite metals, all these metals giving salts of the same typical composition."

In view of these diametrically opposite statements I have taken especial pains to ascertain the true behaviour of the double salt.

17·717 grs. of re-crystallised scandium sulphate, found by analysis to be pure, were dissolved in water in a flask. The calculated quantity of potassium sulphate to unite with the 17·717 grs., and to make a saturated solution in the water added, was then weighed out, and 5 per cent additional potassium sulphate was added to ensure complete saturation. This was dissolved in boiling water, put into the flask containing the scandium sulphate, and water up to 250 cc. was added. The flask was securely corked, and put into a mechanical agitator, where it was shaken continuously for twenty-five hours. At first, when the two salts were mixed, no precipitation of double salt was visible, but after a short time there appeared a fine granular precipitate, which gradually increased as the shaking proceeded.

At the end of twenty-five hours the precipitated double sulphate was filtered off and washed with 100 cc. of cold saturated solution of potassium sulphate. The filtrate was heated, ammonia added, and the whole well boiled. A very slight precipitate of scandia came down which, when filtered, washed, and ignited, weighed 0·414 gr., equivalent to 1·473 grs. of scandium sulphate in solution, the rest of the sulphate being in the insoluble precipitate. It therefore follows that for every 100 parts of the double sulphate 8·32 per cent are soluble and 91·68 per cent are insoluble in the amount of saturated solution of potassium sulphate employed.

The insoluble double salt was washed with boiling water to remove the little excess of potassium sulphate, until its bulk was reduced about one-half. It was then dried and analysed.

My own analytical results agree closely with Nilson's formula. As Cleve distinctly says that the double sulphate is soluble in a saturated solution of potassium sulphate, it is probable he was not working on the true double salt.

The scandia in the aqueous washings was also determined. The total amount of scandia in the washings and precipitate amounted to 4·560 grs., which, added to the 0·414 in the solution, makes 4·974 grs. of scandia. The 17·717 grs. of scandium sulphate contain theoretically 4·981 grs. of scandia, so these results are well within experimental errors.

Scandium Selenate.

$Sc_2(SeO_4)_3,8H_2O$.

Dilute and warm selenic acid easily dissolves scandium hydrate or carbonate, and on evaporating the acid solution over sulphuric acid the selenate crystallises in well-defined rhombic prisms, bright, colourless, and capable of withstanding a temperature of 400° without decomposition, except loss of water.

When heated to 100° for sixteen hours, the 8-hydrate oses 6 molecules of water, and becomes a 2-hydrate,—

$Sc_2(SeO_4)_3,2H_2O$.

Analysis.

1. 16·847 grs. of crystallised scandium selenate, heated to 400°, lost 3·709 grs. of water.
2. 16·847 grs. of the same salt gave on ignition to a full yellow heat 3·485 grs. of scandia, = 2·257 grs. of scandium.
3. 13·913 grs. of the crystallised salt, heated to 100° for sixteen hours, lost 2·293 grs. of water.

	Theory.			Experiment.	
				1, 2.	3.
Sc_2 ..	88·200	13·33		13·39	—
$3SeO_4$.	429·600	64·90		64·60	—
$6H_2O$..	108·096	16·33	21·77	22·01	16·48
$2H_2O$..	36·032	5·44			—
	661·928	100·00		100·00	

The anhydrous selenate, heated to a full yellow heat, loses all the selenic acid, and scandia remains.

Analysis.

13·138 grs. of anhydrous selenate left 3·485 grs. of scandia, = 2·257 grs. of scandium.

	Theory.		Experiment.
Sc_2	88·2	17·03	17·18
$3SeO_4$	429·6	82·97	82·82
	517·8	100·00	100·00

Scandium Nitrate, $Sc(NO_3)_3,4H_2O$.

When hydrated, scandia easily dissolves in dilute nitric acid; after ignition it dissolves with the aid of heat in moderately strong nitric acid. The salt is very soluble in water and in alcohol, and is extremely deliquescent. The acid solution, over sulphuric acid, dries up to a viscous gummy mass, in which appear groups of stellate crystals. A neutral aqueous solution evaporated on the water-bath, on cooling, deposits colourless prismatic crystals.

Scandium nitrate decomposes more easily by heat than the nitrate of any other of the rare earths. The crystals deposited from an almost neutral solution get pasty at 100°, but do not run to liquid. In vacuum at 100° the crystals give off water, and frit together, but do not become liquid. Dried on a water-bath, and heated in a tube immersed in melted paraffin with a thermometer by its side, they show signs of melting at 125°. They commence to liquefy at 150°, and when stirred with a platinum wire the crystals run together and form an opaque white liquid.

When scandium nitrate is partly decomposed by heat, and the melted mass extracted with hot water and well boiled, a basic nitrate then deposits, which is difficult to filter owing to its clogging fineness.

The hydrated nitrate, gradually heated in a silica crucible, fuses and boils, becomes opaque and evolves much water. If the heating is stopped at the proper time, the anhydrous nitrate is left, the whole of the water having been driven off. On continuing the heat nitrous vapours come off, and the liquid becomes clear and liquid like water. Increasing the heat turns the liquid brown, and drives off more nitrous vapours, ebullition becomes sluggish,

the fused mass gets white and opaque, and finally there remains a white residue of scandia.

For analysis the nitrate was thrice re-crystallised and repeatedly dried between blotting-paper.

Analysis.

1. 11·136 grs., gradually heated to full redness, left 2·537 grs. of scandia, = 1·6429 grs. of scandium.
2. 25·978 grs. left 5·914 grs. of scandia, = 3·8297 grs. of scandium.

	Theory.		Experiment.	
			1.	2.
Sc	44·100	14·59	14·75	14·74
$3(NO_3)$ .. .. $4H_2O$	258·094	85·41	85·25	85·26
	302·194	100·00	100·00	100·00

When the above hydrate is dried over a water-bath, it does not fuse, but becomes pasty and anhydrous—

$$Sc(NO_3)_3.$$

Analysis.

1. 38·795 grs. left on ignition 11·449 grs. of scandia, = 7·4141 grs. of scandium.
2. 11·026 grs. of nitrate left on ignition 3·233 grs. of scandia, = 2·0936 grs. of scandium.
3. 14·618 grs. of nitrate left on ignition 4·287 grs. of scandia, = 2·7762 grs. of scandium.

Synthesis.

4. 4·246 grs. of ignited scandia (= 2·7496 grs. of scandium) were covered in a crucible with strong nitric acid and put into a hot oven at 100° for seven hours. All excess of nitric acid disappeared, and a clear oily liquid remained which solidified on cooling. No crystals were seen, but the substance resembled glass. The solid nitrate weighed 14·008 grs.
5. 3·233 grs. of scandia (= 2·0936 grs. of scandium), treated as described in the last experiment, formed 11·026 grs. of nitrate.

	Theory.		Experiment.				
			1.	2.	3.	4.	5.
Sc ..	44·10	19·16	19·11	18·99	18·99	19·63	18·99
$3(NO_3)$	186·03	80·84	80·89	81·01	81·01	80·37	81·01
	230·13	100·00	100·00	100·00	100·00	100·00	100·00

When the hydrated nitrate is dried *in vacuo* at 100° for seventeen hours it becomes a basic nitrate—

$$ScOH(NO_3)_2,H_2O.$$

Analysis.

21·679 grs. of this basic nitrate lost on ignition 14·475 grs., and left 7·204 grs. of scandia, = 4·665 grs. of scandium.

	Theory.		Experiment.
Sc	44·100	21·71	21·52
OH .. .. $(NO_3)_2$ .. .. H_2O	159·044	78·29	78·48
	203 144	100·00	100·00

Therefore the nitrate has lost one equivalent of nitric acid.

When the nitrate is heated for twelve hours in a hot-air oven at a temperature of 120°, a basic nitrate is left of the composition—

$$Sc_2O(NO_3)_4.$$

Analysis.

1. 6·550 grs. left on ignition 2·537 grs. of scandia, = 1·6429 grs. of scandium.
2. 13·698 grs. left on ignition 5·287 grs. of scandia, = 3·4237 grs. of scandium.

	Theory.		Experiment.	
			1.	2.
2Sc	88·20	25·04	25·08	24·99
O .. .. $4(NO_3)$ ..	264·04	74·96	74·92	75·01
	352·24	100·00	100·00	100·00

SCANDIUM FORMATE,

$$(HCOO)_2ScOH,H_2O.$$

Scandium hydroxide dissolves readily in formic acid. The solution is evaporated to expel excess of acid and dissolved in water. It is then allowed to evaporate over sulphuric acid in a desiccator, when it forms hard and lustrous crystals, easily soluble in hot water and in alcohol.

The formate readily decomposes when heated, and at a red heat leaves a white residue of scandia.

Analysis.

8·895 grs. of crystallised formate left on ignition 3·650 grs. of scandia, = 2·363 grs. of scandium.

	Theory.		Experimen
Sc	44·10	26·07	26·57
$(HCOO)_2$ .. .. OH .. .. .. H_2O	125·04	73·93	73·43
	169·14	100·00	100·00

(To be continued).

THE ACTION OF CHLORINE UPON UREA WHEREBY A DICHLORO UREA IS PRODUCED.*

By FREDERICK DANIEL CHATTAWAY, F.R.S.

ALTHOUGH a few of the more familiar substances containing halogen attached to nitrogen, as, for example, nitrogen chloride, have been known for a long period, it is only within the last ten years that such compounds have been systematically studied. They form, however, a group of extraordinary interest, and play an all-important part in many complex reactions; to illustrate this it is only necessary to refer to the substitution of halogen in anilides and to the well-known method for obtaining amines from amides. I have shown in a series of investigations that hydrogen attached to nitrogen in compounds of the most varied characters may be replaced with ease by chlorine or bromine if suitable conditions are observed, and it may be said, speaking generally, that this can always be done, although the nitrogen halogen derivatives produced may react or undergo isomeric change so readily that they can only with difficulty be isolated.

All compounds in which halogen is directly attached to trivalent nitrogen can take part in certain well-defined reactions characteristic of the linkage. One of the most striking of these group reactions is that with hydriodic acid, whereby at the ordinary temperature the halogen is replaced by hydrogen and iodine is liberated quantitatively. This reaction, which may be expressed by the general equation $:NX+2HI=:NH+HX+I_2$, affords an easy method of analysing these compounds.

Such a nitrogen halogen derivative is formed as an intermediate product in a reaction with urea, the course of which has never hitherto been explained although it has

* A Paper read before the Royal Society, June 25, 1908.

received an unusual amount of attention on account of its furnishing a ready method of estimating the quantity of this substance present in a liquid.

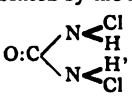
When urea is added to a solution of an alkaline hypochlorite or hypobromite it is at once decomposed, nitrogen and carbon dioxide being set free; if an excess of alkali is used the carbon dioxide is fixed, and the nitrogen, which alone escapes, should thus afford a measure of the amount of urea. The reaction is generally represented by the equation $CO(NH_2)_2+3NaOBr = CO_2 + N_2+3NaBr+2H_2O$. which throws no light upon its nature; indeed, it obscures it, as it makes it appear to be a case of oxidation. It does not even express quantitatively what takes place, for all chemists who have investigated the decomposition have noted that the amount of nitrogen liberated is invariably less than that contained in the urea used. When the operations are carried out under specified conditions this loss of nitrogen, which may amount to as much as half of the total, is very constant, and so can be allowed for by adding a definite fraction of the whole to the gas actually measured. The method can thus be made to give results sufficiently accurate for clinical purposes, and since it is easy to carry out it has received very general application. It is not known what becomes of the nitrogen which does not appear as gas, for all the suggestions that have been made hitherto wholly fail to account for more than a small fraction of the quantity that disappears.

If urea be added instead of to an alkaline solution of a hypochlorite to one acidified by acetic acid, that is to a solution of hypochlorous acid, no gas is evolved, nor is any gas liberated if chlorine itself is passed into a solution of urea in acetic acid. Action, however, takes place in each case, and a nitrogen chloride is produced; for although the hypochlorous acid or chlorine disappears, the resulting solution liberates iodine in large quantity from hydriodic acid.

The isolation of the substance produced from such a solution is not practicable, since the method of extraction by chloroform which often serves for the separation of substituted nitrogen chlorides cannot be used, as the chloro urea formed is easily soluble in water but almost insoluble in chloroform. It is, however, less soluble in water than urea itself, and crystallises out in a pure condition when chlorine in excess is passed rapidly through a cooled sufficiently strong aqueous solution of urea. The action which takes place is represented by the equation $CO<^{NH_2}_{NH_2} + 2Cl_2 = CO<^{NHCl}_{NHCl} + 2HCl$, two only of the four hydrogen atoms of the urea being replaced by chlorine.

This dichloro derivative appears to be the only stable nitrogen chloride which urea is capable of forming. The crystals which separate from the solution of urea have this composition from the first, and chlorine seems to have no further substituting action upon them.

Dichloro urea, having regard to its mode of formation, as well as to the structure of urea itself, has most probably the constitution represented by the formula—



which explains its formation and such of its reactions as have yet been studied.

If this constitution be granted it seems probable that the reaction between urea and chlorine takes place in two stages, and that the amino groups are substituted successively, but the monochloro derivative does not crystallise out, possibly owing to its solubility being not far removed from that of urea itself. Either of two causes may prevent the formation of a ter- or tetrachloro derivative; the hydrochloric acid which is formed in the reaction may prevent that addition of chlorine to the nitrogen which must precede further substitution, or the more highly substituted urea may be hydrolysed so easily that it breaks up as soon as it is formed. Dichloro urea is so much more easily hydrolysed than urea itself that the latter is the more probable cause.

Although two molecules of hydrogen chloride are formed when urea is substituted by chlorine, very little heat is developed; dichloro urea must therefore be an endothermic compound, and might be expected to be highly explosive. When heated, however, it does not explode itself, but decomposes with liberation of nitrogen chloride, which, if heated a few degrees above the temperature at which it is set free, may detonate with great violence.

Dichloro urea gives all the characteristic reactions of a typical nitrogen chloride; for instance, it liberates iodine from hydriodic acid, chlorine from hydrochloric acid, and reacts with alcohol, forming ethyl hypochlorite, urea being in each case re-formed.

It is distinguished from most other substituted nitrogen chlorides by the readiness with which it is hydrolysed in presence of water, nitrogen chloride, carbon dioxide, a little nitrogen and ammonium chloride being formed. If the compound is dissolved in water or kept in a moist atmosphere, this hydrolysis takes place slowly at the ordinary temperature, and becomes very rapid at about 30° C.

It is probable that in this reaction a mono-substituted ammonia is first produced, thus:—

$$CO<^{NHCl}_{NHCl} + 2H_2O = CO<^{OH}_{OH} + 2NH_2Cl;$$

but if so it apparently can only exist momentarily, as nitrogen chloride is at once liberated. The formation of the end products of the reaction can be explained by assuming that this monochloro ammonia at once breaks up into ammonia and nitrogen chloride, $3NH_2Cl = 2NH_3 + NCl_3$, which then to some extent react, forming nitrogen and hydrogen chloride, the latter at once combining with the free ammonia and allowing the remaining nitrogen chloride to escape, as this does not react with ammonium chloride. Both acids and alkalis accelerate the rate of hydrolysis, aud also alter the nature of the end products by hindering or furthering the secondary reaction between the ammonia and the nitrogen chloride.

In the presence of dilute acids the ammonia is at once fixed, and the reaction between it and the nitrogen chloride with its accompanying liberation of nitrogen is prevented; all the chlorine contained in the dichloro urea is therefore liberated as nitrogen chloride. In presence of alkalis, on the other hand, the reaction between the ammonia and the nitrogen chloride goes on to completion, since the hydrochloric acid formed in it is at once fixed; no nitrogen chloride therefore is set free, since twice as much ammonia is formed as is required to decompose it.

The reaction between dichloro urea and a solution of caustic potash is instantaneous; nitrogen is liberated with violent effervescence, the excess of ammonia and the alkaline carbonate formed remaining dissolved in the liquid. The action, which is quantitative, is expressed by the equation—

$$3CO<^{NHCl}_{NHCl} + 12KOH = 3K_2CO_3 + 2NH_3 + 6KCl + 2N_2 + 6H_2O.$$

The behaviour of dichloro urea with alkalis affords an explanation of the course of the reaction which occurs when urea is decomposed either by an excess of alkaline hypochlorite or hypobromite. The urea, is, without doubt, converted into dichloro or dibromo urea (see Note), which is at once hydrolysed in the manner above described. In presence of the excess of hypochlorite or hypobromite, the mono-substituted ammonia formed in the hydrolysis may be further substituted to a greater or less extent, nitrogen being evolved quantitatively only when this takes place under such conditions that the amount of hydrogen attached to nitrogen in the reacting system is always sufficient to react completely with the chlorine attached to nitrogen.

NOTE.—It is possible that in absence of free acid, and

in presence of excess of hypochlorite or hypobromite, a tri- or tetra-substitution product may be produced, the formation and decomposition of which can be formulated easily, but this does not affect the essential character of the reaction, which is one of halogen substitution followed by hydrolysis of the substituted urea and interaction between the resulting compounds.

Preparation of Dichloro Urea.—The preparation of dichloro urea should be carried out at a low temperature and as rapidly as possible, since, in presence of the hydrochloric acid formed at the same time, hydrolysis so readily occurs and so much nitrogen chloride is produced that if it is carried out at the ordinary temperature, or its duration is unnecessarily prolonged, a poor yield is obtained. It is best therefore to work with small quantities, to cool thoroughly, and to pass the chlorine as rapidly as possible. The following procedure gives a good result:—Dissolve 20 grms. of urea in 40 cc. of water, and cool to about −10° C. in a mixture of alcohol and crushed ice. (It is not absolutely necessary to use a freezing mixture, the temperature, owing to the urea dissolving, falls at once to below zero, and if the flask in which the operation is carried out is cooled by tap-water, a moderate amount of the compound can be obtained, but the yield is much better at a lower temperature). Pass in as rapidly as practicable a stream of chlorine made by dropping strong hydrochloric acid upon bleaching-powder. So little heat is evolved in the reaction that with proper cooling the temperature never rises above zero, however rapid the stream of chlorine. Nitrogen chloride is produced from the first, and can be recognised by its characteristic smell, but it remains in solution, the liquid, owing to its presence, becoming bright yellow in colour. After passing the chlorine for a considerable time, white crystals make their appearance, at first usually on the surface of the liquid; these increase in amount till the whole becomes a pulp of thin colourless plates. When these no longer appear to increase in amount, filter off, separating the acid mother-liquor as completely as possible; wash once or twice with a little water, and when as much of the latter as possible has been removed wash the crystals several times with dry chloroform. Dichloro urea is not appreciably soluble in cold chloroform and a good deal of adhering moisture is thus got rid of. Press finally between filter-paper, spread out in a thin layer on a clock glass, and free from the last traces of water by exposing for about half-an-hour over phosphoric oxide in a vacuum.

A further crop of crystals can be obtained by again passing chlorine through the cooled mother-liquor, and this can be repeated as long as any separate. Nothing besides dichloro urea and the products of its hydrolysis are formed in the reaction; if the acid mother-liquor is evaporated down, carbon dioxide is given off and nitrogen chloride escapes in quantity; later, as the liquid becomes concentrated, hydrogen chloride is expelled, and finally ammonium chloride is left.

Although the final result of the action which takes place is the substitution of two atoms of hydrogen by two atoms of chlorine, as in other similar cases, this is without doubt effected by the addition of four atoms of chlorine to the nitrogen followed by the elimination of two molecules of hydrogen chloride.

The yield of dichloro urea is not very large, so much being hydrolysed during the process; in a well conducted experiment it reaches about 25 per cent of the theoretical, the weight of pure dry product obtained amounting as a rule to rather more than half the weight of the urea used. The loss, although largely due to the hydrolysis of the compound, is much increased by the circumstance that dichloro urea is very soluble in water, and consequently a considerable amount does not crystallise out from the mother-liquor.

Dichloro urea reacts with an aqueous solution of hydriodic acid in the way characteristic of nitrogen chlorides; urea is reformed and iodine quantitatively liberated. It was analysed by taking advantage of this reaction; a known weight was added to an excess of a solution of potassium iodide made acid by acetic acid and the iodine liberated estimated by a standard solution of sodium thiosulphate.

0·5112 grm. liberated iodine = 158·6 cc. N/10 I. Cl, as :NCl = 54·99 per cent. $CON_2H_2Cl_2$ requires Cl, as :NCl = 54·96 per cent.

Dichloro urea cannot be kept for any length of time without change. When damp it hydrolyses exactly as when dissolved in water. To obtain it pure, therefore, it must be dried very rapidly over phosphoric oxide in a vacuum. When only freed from adhering water as far as possible by a pump, it is slowly hydrolysed by the retained moisture even though placed in a desiccator over strong sulphuric acid, and this often occurs to such an extent that the mass becomes quite yellow from the nitrogen chloride mechanically retained by the crystals. To show the amount of hydrolysis which takes place under these conditions a quantity of the pure substance freed from all but adhering water thus was kept over strong sulphuric acid, and analysed from time to time, the nitrogen chloride formed being allowed occasionally to escape. In two days the percentage of chlorine as :NCl had fallen to 43·79, and in seven days to 13·33.

Even when dried and kept over phosphoric oxide it slowly decomposes, nitrogen chloride being evolved. A quantity of the pure dry compound thus preserved in a vacuum over phosphoric oxide was analysed at intervals; after twelve hours the percentage of Cl as :NCl had fallen to 54·67, after two days to 52·97, and after nine days to 47·87 per cent.

When an aqueous solution is kept at the ordinary temperature the dichloro urea is slowly hydrolysed; carbon dioxide, a little nitrogen, and nitrogen chloride escaped, and ammonium chloride remains in solution. Hydrolysis by water takes many days to complete unless the aqueous solution is warmed, when it is much more rapid; at about 20° bubbles of gas are freely evolved and rapid effervesence sets in at about 40°, the nitrogen chloride escaping without explosion.

Dichloro urea prepared as described above is a soft white crystalline powder with a pearly lustre. Under the microscope it is seen to consist of thin transparent plates somewhat irregular in shape. It can be re-crystallised from water, in which it is easily soluble and from which it separates in similar but larger plates, by cooling an aqueous solution saturated at about 15° C., though with some loss owing to hydrolysis. Its aqueous solution is at first colourless but becomes yellow as hydrolysis proceeds, owing to the liberation of yellow nitrogen chloride which remains dissolved in the water. It is easily soluble in alcohol and ether, very slightly soluble in chloroform, and insoluble in petroleum ether. When its alcoholic solution is heated it reacts in the way characteristic of nitrogen chlorides, urea is reformed, and ethyl hypochlorite is produced, the latter very easily breaking down into aldehyde and hydrogen chloride.

Its behaviour with acids is complicated by the circumstance that it is so readily hydrolysed; for example, when it is added to strong hydrochloric acid, though chlorine is rapidly given off the amount of urea reformed is not very large. When placed in cold strong sulphuric acid it is hydrolysed and nitrogen chloride is liberated. The latter decomposes into its elements if the liquid be heated, its characteristic smell disappearing and being replaced by that of chlorine.

Dichloro urea is a compound of a marked acid character, it has an acid taste recalling that of hypochlorous acid and its aqueous solution strongly reddens litmus-paper, which only becomes bleached after the lapse of some minutes. It acts very corrosively upon the skin, staining it yellow and destroying the tissues.

When heated, dichloro urea melts with decomposition at about 83°, nitrogen chloride being liberated. Although the substance appears not itself to explode, if the temperature

of the bath in which the melting-point is being taken is allowed to rise at all rapidly a few degrees above this point, the nitrogen chloride set free in the melting-point tube may explode with considerable violence.

It can be decomposed without danger by throwing it into a porcelain dish heated to 100° on a water-bath in quantities of not more than about half a grm. at a time. The white compound fuses and gives off nitrogen chloride as a yellow vapour which escapes quietly unless, through too large a quantity of material having been decomposed at once, some of the vapour is mechanically retained as bubbles in the semi-fused residue, and thus becomes heated to the temperature at which it explodes. Hydrogen chloride is not set free when dichloro urea is thus decomposed by heat.

When an aqueous solution of the compound is added to a solution of caustic soda, vigorous effervescence, due to escape of nitrogen, occurs. The liquid remaining contains ammonia and potassium carbonate, the volume of carbon dioxide liberated when this is treated with an acid being to the volume of nitrogen previously liberated in the ratio of 3 to 2. One-third of the nitrogen contained in the dichloro urea used is found as ammonia.

The investigation of dichloro urea, which is an extremely reactive body and promises to be of considerable use in organic synthesis, is being continued.

The thanks of the author are due to Dr. Baker for allowing him to use the Christ Church Laboratory, where this work has been carried out.

NOTE ON

THE FORMATION OF FORMALDEHYDE IN SOLUTIONS OF CANE-SUGAR, AND ITS BEARING ON HEHNER'S TEST FOR FORMALDEHYDE IN SACCHARIN MIXTURES.

By A. ALEXANDER RAMSAY.

TRILLAT has shown that on heating cane-sugar, traces of formaldehyde are given off even at 125° C. ("The Formation of Formaldehyde in the Caramelisation of Sugar," *Bull. Soc. Chim.*, 1906, xxv., 681). At 150° C. the aldehyde is liberated in greater quantity. Analysis of the vapours emitted at 200° C. showed the presence of 0·2 to 5·7 per cent formaldehyde, and the residual caramel contained up to 0·27 per cent of polymerised formaldehyde, probably in the state of trioxymethylene. The amounts produced were found to vary with the method of heating, the nature of the vessel, and the purity of the sugar.

The object of the present note is to draw attention to the fact that the production of formaldehyde takes place in minute quantities when cane-sugar and water are heated together from 100—103° C. This fact renders the Hehner test (*Analyst*, 1896, xxi., 95) unreliable as an indication of the addition of formalin to jam or saccharin beverages, since formaldehyde is produced in the manufacture of jam. Moreover, the act of distilling a saccharin liquid results in the formation of formaldehyde, even if it were not originally present.

The following experiments were performed :—

1. 100 grms. pure cane-sugar was dissolved in 5 ozs. cold distilled water, heated for half an hour, and distilled. The distillate gave the reaction for formaldehyde with Hehner's test (*loc. cit.*). The boiling-point of the residue in the retort was 103° C.

2. 50 grms. pure cane-sugar was dissolved in 5 ozs. cold distilled water in a glass retort and heated till the mixture boiled, and distilled. The distillate which was obtained during the first twenty minutes was collected and marked *a*. The receiver was changed, and the distillation continued for about twenty minutes longer, the distillate collected and marked *b*. The liquid remaining in the retort was diluted with cold distilled water to 5 ozs., and again heated and distilled—collecting the distillate separately and marking it *c*. When the distillation was finished the sugar solution in the glass retort was crystal clear, had no yellow tinge, boiled at 101—101·5° C., and gave a strong reaction for formalin.

The three portions of distillates marked *a*, *b*, *c* were tested for formaldehyde by Hehner's test, and all gave the reaction, though faintly. The remainder of portion marked *a* was re-distilled, and the first 5 cc. caught and marked *a'*. This gave a strong reaction for formaldehyde both by Hehner's test and Pilhastry's test (Cohn, "Tests and Reagents").

The remaining portions of distillate marked *b* and *c* were similarly treated, the first 5 cc. of each caught and marked *b' c'* respectively. Both *b'* and *c'* gave the Hehner reaction for formalin, *c'* less strongly than *a'* or *b'*.

The foregoing taken with Trillat's note, explains the occurrence of formaldehyde in jam made from pure fruit, cane-sugar, and water. Jam was prepared from pure fruit and pure cane-sugar as follows :—

Peach.—1 lb. fruit, 1 lb. sugar, 3½ ozs. water, boiled about two hours.

Orange.—1 lb. fruit, 2 lbs. sugar, 13 pints water, boiled about two hours.

Quince.—1 lb. fruit, ¾ lb. sugar, 1 pint water, boiled about two hours.

Quince Jelly.—1 lb. fruit, 1 lb. sugar, 1 pint water, boiled about two hours.

Unfortunately at the time these jams were prepared the boiling-points of the various mixtures was not noted. The temperature was certainly never above that reached in making jam on the manufacturing scale, and there was no possibility of caramelisation.

On examining the foregoing four jams for formalin (by distilling a portion faintly acidified and testing the distillate) reactions were obtained in all cases—in the case of the orange jam the reaction was particularly strong.

Further investigation as to the amounts of formaldehyde present are being carried out, and the results will be embodied in a future paper. The experiment also shows the unsuitability of the method of examining jams for formaldehyde, by dissolving 50 grms. sample in 250 cc. water as recommended by Leach ("Food Inspection and Analyses"), acidifying with phosphoric acid, distilling, and testing the first 30 cc. distillate for formalin. After 125 cc. distillate has been obtained, further distillation yields a distillate containing still larger quantities of the aldehyde.

The presence of formaldehyde in various foods has been noted by Perrier ("Presence of Formalin in Certain Foods," *Comptes Rendus*, 1906, cxliii., 600),, who finds in cider 0·2 to 0·33 parts formaldehyde per 100,000, and in bacon, ham, sausage, herring, &c., amounts varying from 0·03 to 0·003 parts per 100,000. In jams examined by the author the amounts of formaldehyde have varied from 1·3 to 14 parts per 100,000.

Experiment 2 mentioned previously was repeated with the object of applying other confirmatory tests for the formaldehyde present, by the Gayon-Saglien-Mohler reagent (M. X. Rocques. "Analysis of Alcohol and Brandy," *Rev. Internat. de fals. and d'Anal. des Matieres Alimentaires*, April, 1907, p. 49), Pilhastry's reagent (Cohn, "Tests and Reagents"), Hehner—aqueous phenol (*Ibid.*, p. 121), and Hehner—commercial sulphuric acid (*Ibid.*, p. 121). Reactions clear and well defined were obtained by both Hehner's tests and a faint reaction with Pilhastry's reagent, but no reaction with the Gayon-Saglien-Mohler reagent.

While carrying out these tests I found Hehner's milk-sulphuric acid test most delicate ; 1 part formalin in four millions gave a clear reaction in two minutes, and up to 1 in ten million on standing a little longer, provided the milk is added to the solution of formaldehyde immediately before the addition of the sulphuric acid. The aqueous phenol and sulphuric acid reacted up to 1 part in four millions. Pilhastry's reagent seems to detect only 1 part

in half a million, and the Gayon-Saglien-Mohler reagent about 1 part in a quarter of a million.

It has also been shown by Forster that furfuraldehyde is produced in small quantities by boiling sugar with water (*Ber.*, xv., 230), but in the experiments detailed above I find no furfural reaction, only formaldehyde. In the case of furfural in dilute aqueous solution with Hehner's test (milk-sulphuric acid), at the junction of the two liquids a faint pink zone is formed, which darkens, forming a decided green colour which deepens on standing. With Hehner's aqueous phenol and sulphuric acid test applied to furfural, a purple black band forms at junction of the liquids which deepens in colour on standing and increases in size. These reactions are quite dissimilar to the reactions of formaldehyde.—*Journal and Proceedings of the Royal Society of N. S. Wales*, vol. xli., p. 172.

THE PRESENT STATE OF OUR KNOWLEDGE OF RADIO-ACTIVITY.*

By A. DEBIERNE.

(Continued from p. 273).

II.

IT will be observed that though the numbers obtained for thorium and uranium are very large and comparable with the intervals of time reckoned in geological transformations, the life of radium, on the contrary, is comparatively short; its existence on the earth's surface could not be explained unless the destruction of it was compensated by a continuous production, and this production must take place at the expense of the substances which accompany it in minerals. The fact, proved in Mdme. Curie's first experiments, that only minerals which contain uranium or thorium are appreciably radio-active, and the fact that radium is extracted from uranium minerals, lead us to suppose that it is uranium which produces radium, and that in radio-active transformations the radium series is a continuation of the uranium series.

Researches on the radio-active minerals have disclosed an important argument in favour of this view. Boltwood has determined the amount of uranium in a large number of radio-active minerals obtained from various places in Europe and America, and at the same time he has measured the ionisation produced at the end of three hours in a given container by the emanation set free by these minerals when they are dissolved in an acid.

As, up to the present, radium emanation is the only emanation known which has a fairly long life, there is reason for thinking that it alone exists in gases extracted from minerals some hours after extraction. Moreover, the ionisation produced in the same container by the emanation set free by a given quantity of radium has been determined, and hence the proportion of radium contained in the minerals could easily be calculated. Applying various corrections for the liberation of emanation by the minerals before they were treated, Boltwood found that the ratio between the quantity of radium and of uranium is a constant for all uranium minerals. This result is confirmed by the experiments of McCoy and Eve, in which the intensity of the radiation of the minerals was measured, taking into account certain peculiarities of the radiation of radium. Thus the methods of determining radium are indirect, but the agreement of the results is such that it is difficult to entertain any doubts concerning the actual existence of this constant ratio of radium to uranium in minerals. (However, Danne has shown that certain pyromorphites containing considerable amounts of radium do not contain uranium, but this anomaly can easily be explained as a chemical separation effected by the action of water).

This fact, which in itself indicates a connection between radium and uranium, may be explained as follows:—

* *Revue Générale des Sciences.* 1908, xix., 730.

If radium is produced by uranium, and if the destruction of radium is rapid compared to that of uranium, it is easy to see that the quantity of radium in equilibrium is directly proportional to the quantity produced by the uranium in a given time; that is to say, to the total quantity of uranium. It must thus be supposed that uranium has a very slow rate of decay, in order to explain its presence on the surface of the earth. Other considerations point to a life of 5×10^9 years.

If the production of radium from uranium were direct, according to the above data, the phenomenon would be of sufficient magnitude to be established directly by experiment.

Different experiments have been arranged by Soddy. He first obtained a positive result which, however, was much smaller than that foretold by theory; his later experiments gave so low a result that it might be due to experimental errors; in fact, up to the present time the formation of radium by uranium has not been proved by direct experiment.

To explain this result and yet ascribe to radium the relatively short life, demanded in accordance with Rutherford's hypotheses, it may be supposed that radium is not the direct product of uranium. Then if an intermediate term exists, having a very long mean duration of life, the formation of radium from pure uranium could not be proved until a large enough quantity of this intermediate product had been formed and destroyed; this would explain the negative result of the experiments on the direct formation of radium from uranium.

Recent experiments by Rutherford and Boltwood verify this supposition. Some time ago I showed that compounds of actinium always liberate very small quantities of an emanation with a slow rate of decay, which I afterwards found to be radium emanation; thus actinium might be supposed to be the intermediate substance. Boltwood and Rutherford first thought this; moreover, they proved a slow increase in the quantity of radium emanation in certain specimens of actinium. However, they recognised that this production of emanation, which apparently involves that of radium, is due to a substance differing from actinium and accompanying it in fairly large quantities in the specimens used by Rutherford and Boltwood. This new substance, which has been prepared only in a very dilute state, Boltwood called ionium. (The proportion of radium emanation, liberated by my specimens of actinium, is much less than the proportion found by Rutherford in a specimen obtained from Giesel). Thus, summarising, we may say that if the direct production has not been shown experimentally the succession is very probable, and the negative result can easily be explained.

According to these experiments, uranium and radium would form a single series; but, as far as we know, thorium and actinium would form separate series, unrelated to one another and to the other series.

III.

Radio-active transformations may be accompanied by different secondary phenomena. We have already seen that the rays emitted by radio-active substances produce the phenomena of ionisation, phosphorescence, and photographic action. Chemical actions have also been demonstrated. The first action to be observed was the transformation of oxygen into ozone under the influence of radium emanation (Pierre Curie and Mdme. Curie). By the action of the radiation from radium, white phosphorus is transformed into red phosphorus, iodine is set free from iodic acid, &c.

The most remarkable chemical action is the decomposition of water into hydrogen and oxygen; it was first observed by Giesel with radium solutions; with a solution containing 1 decigrm. of radium bromide little bubbles of gas can easily be seen forming continuously in the midst of the solution. Ramsay in recent experiments obtained the same decomposition by the action of radium emanation; he also proved that the quantity of oxygen obtained is smaller than that corresponding to the formula H_2+O.

I observed that actinium solutions exhibit the same phenomenon, and that the liberation of gas in a solution is perfectly regular and proportional to the time. In recent experiments, which have not yet been published, I have found that the decomposition of water is due only to the penetrating radium rays. This is a very interesting result, for I think it is the first time that such a reaction has been caused by the action of a radiation.

The rays of radio-active substances and particularly those of radium have the power of colouring certain substances, glass, porcelain, alkaline salts, barium platinocyanide, fluorspars, quartz, diamond, &c. These colorations, which may be very intense, seem to be the same as those which are caused by the cathode rays. Many hypotheses concerning the nature of these colorations have been suggested, but very little is actually known about them. By the action of cathode rays on common salt metallic sodium is formed in the mass, and can be separated by distillation; hence it has been suggested that the colorations may be due to the presence of metallic particles. However, I think that the colorations cannot generally be due to metallic particles, and that the reaction must occur in two stages, the first being analogous to that observed with silver salts on a photographic plate, before they are reduced to the state of metallic silver.

I have suggested a theory in which these coloration phenomena are connected with the phenomena of phosphorescence and thermoluminescence; it may be mentioned that the presence of very small quantities of impurities has a marked influence on the nature of the coloration, and this is to be ascribed to the influence of the impurities on the phenomena of phosphorescence.

Chemical actions under the influence of radio-active transformations have been but little studied; it seems, however, very probable that they are very frequently produced, either by the action of the rays or of the centres of radio-activity. Mdme. Curie, who showed some time ago that the condensation of gases is produced in presence of the emanation, has observed that these phenomena are accompanied by chemical reactions. Probably radio-active substances are very energetic agents in producing chemical reactions, but the small quantities dealt with render the study of the subject very difficult.

Recent experiments by Ramsay and Cameron go to show that much more energetic actions may be induced by the aid of radio-active substances. In fact these investigators have reported that under the influence of radium emanation ordinary elements may be dissociated and transformed into other elements. The experiments are as follows:—If large quantities of radium emanation are allowed to act on distilled water, besides the oxygen and hydrogen resulting from the decomposition of the water, a small quantity of neon is obtained in place of the helium which was formed in previous experiments with the dry emanation. When this emanation is allowed to act on a solution of a copper salt the gas obtained is argon; moreover, in the solution, after the separation of the copper, an appreciable amount of sodium and potassium salts and a small quantity of lithium salts can be detected. In control experiments performed without emanation or without copper salts so large a quantity of alkaline salts was not obtained, nor was the presence of lithium detected.

Ramsay concludes from these experiments that copper undergoes a disintegration under the action of the emanation, and that potassium, sodium, and lithium are the products of this disintegration.

These results are too important to be accepted without a rigorous experimental proof, and it must be noted that such a demonstration is extremely difficult in the present case. As a matter of fact, these experiments are far less conclusive than those which show the formation of helium by radium. For the element helium does not exist in the air nor in the substances which are present during the experiments. In many blank experiments performed in absence of radium under different conditions, I have never observed the formation of helium. The brilliant and very characteristic spectrum of this element makes it quite easy to detect its formation with certainty; finally, in my experiments I have been able to establish an almost perfect proportionality between the duration of the action of the radium and the amount of helium obtained. Thus many experiments have confirmed the discovery of Ramsay and Soddy, and Giesel has recently confirmed my experiments on the formation of helium by actinium.

On the contrary, in Ramsay and Cameron's recent experiments the elements, which have been obtained by the action of radium emanation are among those which might have been accidentally introduced in the course of the different operations. Neon and more particularly argon are contained in appreciable amounts in air, and a trace of air left in the apparatus might be revealed at the end of the experiment by the presence of these gases, for oxygen is absorbed in the manipulations, and nitrogen might also be eliminated by combining with oxygen under the influence of the emanation. In confirmatory experiments which I performed I always obtained only a small quantity of argon (moreover, this quantity is much smaller than the quantity of helium formed by radium or actinium).

In the experiments with copper salts, as Ramsay himself pointed out, the presence of sodium and potassium might be due to an action on the glass apparatus which was used in the experiments; but we have seen that the proportion of these elements is considerably greater than in the blank experiments, carried out either with the emanation alone or with the copper salt alone. It is not impossible to imagine that the glass of the vessels is rendered peculiarly liable to attack by the simultaneous presence of the emanation, with all the radiations which accompany its destruction, and which may produce a pulverisation of the glass and of the acid solutions of the copper salts.

At first sight it would seem much easier to demonstrate the production of lithium. However, the quantity obtained is much smaller than that of the compounds of sodium and potassium, about 1/10,000 according to Ramsay and Cameron's statements, and the suggestion that the glass it attacked is still feasible; for although an appreciable amount of lithium in glass has not been detected by analysis, it is not impossible that a proportion of 1/10,000 of lithium might occur in the alkalis of the glass; lithium is known to be very widely distributed in nature, especially in the materials which might be used for the preparation of glass. Finally, the unexpected discovery has been made that lithium is a constant impurity in copper salts, and Ramsay was only able to obtain a copper salt free from lithium with very great difficulty.

The formation of lithium has been the subject of many researches on the radio-active minerals. McCoy in America and Mlle. Gleditsch in the laboratory of Mdme. Curie in France, have found independently that all radio-active minerals contain a small quantity of lithium, and and this result seems at first to be an argument in favour of Ramsay's conclusions. But Mlle. Gleditsch, by determining the lithium and also the copper in active minerals, has proved that the quantity of lithium is very small, and that there is no relation between it and the radio-activity, nor between it and the amount of copper present. In particular the mineral which seemed to fulfil best the conditions for the formation of lithium, chalcolite, a phosphate of uranium and copper, which is strongly radio-active and contains the largest proportion of copper, is also the mineral which contains the least lithium. And even the presence of this small amount of lithium might be attributed to the fact that the mineral might contain a small quantity of gangue which contains an appreciable amount of lithium. As Ramsay has pointed out, this result is not an argument absolutely contrary to the formation of lithium, for the latter might depend upon external circumstances not realised in chalcolite. However, seeing that radio-active transformations appear to be absolutely unaffected by external conditions, it is impossible to conceive what the circumstance could be which would hinder the formation of lithium in chalcolite. The

result of the researches on minerals is thus on the whole unfavourable to the idea of the transformation of copper .nto lithium under the action of the emanation.

Thus, in spite of Ramsay's great scientific reputation and the confidence which we feel in his skill as an experimenter, and in spite of his discovery of the formation of helium which has been completely confirmed, I think that we must wait for fresh researches before forming a definite opinion concerning these new results.

It is hardly necessary to point out that if these results are confirmed they will be of fundamental importance for the development of chemical science in general and of researches on radio-activity.

In reality these new transformations are entirely different from those which are assumed in radio-active transformations, and also from the production of helium as it is explained according to our present theories. Thus radio-active transformations occur spontaneously, and we cannot by any means whatever start or stop them, nor even exert any influence over the rate at which they occur; we watch this evolution of the elements as powerless spectators who cannot modify it in any way. But in Ramsay's experiments the transmutations were caused artificially by bringing together two elements, emanation and copper, which are capable of reacting, exactly as in any chemical reaction. This marks a very decided progress, and I sincerely hope that it will be realised.

It is also probable that the present theories of radio-activity will undergo some modifications, for if radium emanation causes atomic transmutations in the substances which accompany it, it is conceivable that these substances may intervene in the radio-active transformations, and that the helium, for example, does not come from the radio-active atom but from the substances which may accompany it.

(To be continued).

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 265).

Confirmatory Experiments and References (continued).

Condition of Vanadic Acid in Solution.—See Düllberg (*Zeit. Physik. Chem.*, 1903, xlv., 129—181). He considers that in a solution of the composition of Na_3VO_4, which reacts strongly alkaline, the salt is largely hydrolysed according to the reaction $2Na_3VO_4 + H_2O = 2NaOH + Na_4V_2O_7$, but his evidence is not convincing. On adding HCl slowly the results indicate the presence of a tetrabasic acid, $H_4V_6O_{17}$. This is a strong acid, two of the hydrogens splitting off nearly completely in dilute solutions, and the third one to a considerable extent. The yellow colour is probably due to the ion $HV_6O_{17}\equiv$. The transition of one form of vanadic acid into another takes place readily, the final equilibrium state being reached fairly quickly. Conductivity and freezing-point determinations make it probable that the formula of sodium metavanadate is $(Na+)_3V_3O_9\equiv$.

*P. 58*b*, N. 1: *Partial Decomposition of* H_2O_2 *in the* $NaHCO_3$ *Treatment.*—A mixture containing 100 mgrms. U as $UO_2(NO_3)_2$ and 100 mgrms. V as Na_3VO_4 was treated by P. 52 and *P. 58*a*, and then was acidified with HNO_3; the acid solution before adding the $NaHCO_3$ was red owing to the presence of pervanadic acid, indicating the presence of much H_2O_2; on neutralising with $NaHCO_3$ it became yellow; and the final HNO_3 solution was also pale yellow. The solution was tested for H_2O_2 with a titanium solution; a distinct colour was obtained. These results show that much but not all of the H_2O_2 had been decomposed.

*P. 58*b*, N. 2: *Detection of Chromic Acid in the Presence of Uranium and Vanadium by* H_2O_2.—A mixture containing 1 mgrm. Cr, 100 mgrms. U, 100 mgrms. Zn, and 100 mgrms. Al as nitrates, and 100 mgrms. V as Na_3VO_4 was treated with NaOH and Na_2O_2 by P. 52; a deep red solution resulted and there was no residue. The solution was made acid with HNO_3, being cooled carefully during the process. Just enough acid was added to dissolve the precipitate that formed. To about one-fourth of the solution in a test-tube was added a little ether and about 1 cc. H_2O_2; the ether layer became blue. The water layer was red before H_2O_2 was added, showing the presence of pervanadic acid. The experiment was repeated with 2 mgrms. Cr, 100 mgrms. U, and 100 mgrms. V; the result was the same. After testing for chromium the remainder of the solution was treated by *P. 58*a*, the solution was cooled, made just acid with HNO_3, and one-tenth of it tested for chromic acid with H_2O_2; a good blue colour was obtained. The remainder of the solution was made somewhat more strongly acid and evaporated to half its volume; the test for chromic acid was then made with half the solution; no blue colour was obtained, showing that the chromic acid had been reduced on boiling.

Action of H_2O_2 *on Uranium Salts.*—To 10 mgrms. U as $UO_2(NO_3)_2$ in 20 cc. were added 1 cc. HNO_3 (1·20), 1 cc. 3 per cent H_2O_2, and a little ether; the yellow colour of the solution did not change, and the ether layer remained colourless.

*P. 58*b*, N. 4: *Precipitation of Chromate by Lead Nitrate.*—Several solutions each containing 0·5 mgrm. Cr as K_2CrO_4, 2 grms. $NaNO_3$, and 1 grm. $NaHCO_3$ in 100 cc. or 70 cc., were made distinctly acid with HNO_3, shaken to expel CO_2, and neutralised exactly with NaOH; varying amounts of HNO_3 (1·20) were added, and finally known volumes of a 20 per cent lead nitrate solution. The results are shown in the following table:—

Initial volume. Cc.	Volume of HNO_3 (1·20). Cc.	Volume of lead nitrate solution. Cc.	
100	1·5	10	Precipitate in 1 or 2 mins.
100	2·5	20	Precipitate in 3 or 4 mins.
100	5·0	30	No precipitate in 30 mins.
70	1·5	10	Precipitate in 2 or 3 mins.
70	2·5	10	Precipitate very slowly.
70	2·5	20	Precipitate in 3 to 5 mins.

The last two experiments were repeated with 6 grms. $NaNO_3$ (instead of 2 grms.); only very small precipitates were obtained in half an hour, even with 20 cc. $Pb(NO_3)_2$ solution, showing that the presence of much $NaNO_3$ prevents the precipitation of $PbCrO_4$.

Behaviour of Vanadate and Uranate towards Lead Nitrate.—Several solutions, each containing 100 mgrms. V as Na_3VO_4, 2 grms. $NaNO_3$, and 1 grm. $NaHCO_3$, in 100 cc. were acidified with HNO_3, shaken, and then neutralised exactly with NaOH; 1·5 to 3 cc. of HNO_3 (1·20) and 10 to 30 cc. of a 20 per cent lead nitrate solution were added, and the mixture allowed to stand thirty minutes; with 3 cc. HNO_3 there was no precipitate even with 30 cc. $Pb(NO_3)_2$; with 1·5 and 2 cc. HNO_3 there was none with 10 cc. $Pb(NO_3)_2$; but a small one with 20 cc., which was however very slight when 2 cc. HNO_3 were used. The experiments were repeated with an initial volume of 70 cc.; the results were nearly the same, except that the precipitate when formed were somewhat larger.

A solution containing 100 mgrms. U as $UO_2(NO_3)_2$, 2 grms. $NaNO_3$, and 1 grm. $NaHCO_3$ in 100 cc. was neutralised with HNO_3, and an excess of 1 cc. HNO_3 (1·20) was added; finally 20 cc. 20 per cent $Pb(NO_3)_2$ were added; no precipitate separated in several hours.

Precipitation of Aluminium, Beryllium, or Lead by Sodium Phosphate in *P. 58*c*.—0·5, 1, and 2 mgrms. Al as nitrate, in solutions containing 2 grms. $NaNO_3$, 2 grms.

* From the *Journal of the American Chemical Society*, xxx., No. 4.

NH_4NO_3, and 5 cc. 30 per cent acetic acid in a total volume of 100 cc. were heated to boiling; flocculent white precipitates separated in each case, the results showing that this is a very good method of estimating small amounts of aluminium.

The experiments were repeated with 0·5, 1, and 2 mgrms. Be as chloride; flocculent white precipitates resulted except in the experiment with 0·5 mgrm. The filtrate in the experiment with 2 mgrms. was estimated to contain about 0·5 mgrm. Be.

The experiments were repeated with 1 and 2 mgrms. Pb as nitrate; a distinct precipitate was obtained with 2 mgrms. but only a very small one with 1 mgrm. Pb.

Oxidation of Hypovanadic Acid by Bromine.—50 mgrms. V as Na_3VO_4 and 1 grm. Pb as $Pb(NO_3)_2$ were dissolved in 2·5 cc. HNO_3 (1·20) and 100 cc. water; the mixture was saturated with H_2S in the cold, filtered, and the filtrate was boiled; the cold solution had a blue colour, but on boiling sulphur separated and the colour became deeper. The sulphur was filtered off, bromine water added until the bromine odour was distinct after shaking, and the mixture was boiled; the colour was still blue. A small excess of bromine was again added; the solution was still blue. The experiment was repeated except that several drops liquid bromine were added, and the mixture shaken, and allowed to stand two or three minutes; the bromine was then boiled off; the solution was yellow. The filtrate was treated by *P. 58*c*; no precipitate separated.

Behaviour of Vanadyl Salts and Vanadates towards Sodium Phosphate.—50 mgrms. V as Na_3VO_4 and 1 grm. Pb as $Pb(NO_3)_2$ were dissolved in 2·5 cc. HNO_3 (1.20) and 100 cc. water; the mixture was saturated with H_2S, filtered, boiled, and filtered again. The blue filtrate was treated by *P. 58*c*, 2 grms. $(NH_4)_2SO_4$ and 2 grms. Na_2HPO_4 being added; on warming, a large, flocculent, bluish white precipitate separated. The experiment was repeated except that the vanadyl salt was first oxidised with liquid bromine as described in P. 58*b*; no precipitate separated in *P. 58*c*.

*P. 58*c*, N. 1: *Precipitation of Uranyl Ammonium Phosphate.*—See Kern. (*Journ. Chem. Soc.*, 1901, xxiii., 705—10).

Detection of Small Amounts of Uranium.—0·3, 0·5, and 1 mgrm. U as $UO_2(NO_3)_2$ were dissolved in separate experiments in 100 cc. water containing 5 cc. 30 per cent acetic acid and 3 grms. $NaNO_3$, and treated by P. 58*c*; white flocculent precipitates separated in each case, but that with 0·3 mgrm. was very small. The precipitates were filtered off and tested by P. 58*d*; in the experiment with 0·3 mgrm. U the ferrocyanide test failed, but was very satisfactory in the other two cases. The experiment with 0·3 mgrm. U was repeated except that 0·5, 4, and 6 grm. respectively $(NH_4)_2SO_4$ (instead of 2 grms.) were added; very small precipitates of uranyl ammonium phosphate were obtained in each case, but the ferrocyanide test failed. This shows that there is no advantage in adding more than 2 grms. $(NH_4)_2SO_4$. The experiments with 0·5 and 1 mgrm. U were repeated, except that no ammonium salt was added; with 1 mgrm. the result was satisfactory, but with 0·5 mgrm. the confirmatory failed.

The first experiments with 0·3, 0·5, and 1·0 mgrm. U were repeated in a volume of 40 cc. instead of 100 cc.; a distinct phosphate precipitate resulted in each case, and the ferrocyanide test was satisfactory, even with 0·3 mgrm. With 0·2 mgrm. a small phosphate precipitate formed, but the confirmatory test failed. The experiment was repeated with 1·0 mgrm. U, except that 0·3 grm. $Na_2HPO_4.12H_2O$ was added instead of 2 grms.; the confirmatory test failed, showing that a large excess of Na_2HPO_4 is necessary. The experiment with 0·3 mgrm. U was repeated except that 10 cc. acetic acid was used instead of 5 cc.; the confirmatory test failed, showing that a large excess of acetic acid makes the phosphate precipitation less complete.

Separation of Uranium and Vanadium by Phosphate.—100 mgrms. U as $UO_2(NO_3)_2$, 50 mgrms. V as Na_3VO_4, and 1 grm. NH_4NO_3 were dissolved in a few drops HNO_3 and 30 cc. water. The mixture was neutralised with NH_4OH; a large pale precipitate of uranyl ammonium vanadate separated. 10 cc. 30 per cent acetic acid were added; the precipitate did not dissolve. 5 cc. 10 per cent ammonium phosphate were added, the mixture was heated to boiling, allowed to stand for fifteen minutes, and filtered. The filtrate was tested for vanadium by P. 58*e*; a large quantity was found. The precipitate was washed with dilute NH_4NO_3 solution, dissolved in dilute hydrochloric acid, and the phosphate precipitation repeated; less than 1 mgrm. V was now found in the filtrate. The phosphate precipitate was treated in the same way; less than 0·2 mgrm. V was found in it. The complete experiment was repeated except that the mixture containing the uranium ammonium vanadate precipitate and the acetic acid was heated to boiling, cooled, and allowed to stand over night before the phosphate was added; the result was the same, showing that the vanadium passes into the filtrate even when it is first precipitated in combination with the uranium.

*P. 58*c*, N. 4: *Precipitation of Uranyl Hydrogen Phosphate.*—See Kern (*Journ. Am. Chem. Soc.*, 1901, xxiii., 705).

*P. 58*d*, N. 1: *Behaviour of Uranyl Salts towards Potassium Ferrocyanide.*—Known amounts of $K_4Fe(CN)_6$ solutions were added to solutions containing 0·5 mgrm. U as $UO_2(NO_3)_2$, 1 cc. HCl (1·12), and 10 cc. nearly saturated NaCl solution; with 4 cc. of a 3 per cent ferrocyanide solution a brown colour appeared only after forty minutes, with 10 cc. a brown precipitate separated in about ten minutes, while with 10 cc. of a 10 per cent ferrocyanide solution a precipitate separated in a minute or two, showing that a large excess of $K_4Fe(CN)_6$ is needed to make the test delicate. The experiment was repeated except that 0·5 cc. HCl (1·12) was added instead of 1 cc., 10 cc. 3 per cent ferrocyanide solution being added; a brown precipitate separated in a minute or two. This experiment was repeated with 0·1 and 0·05 cc. HCl (1·12); brown precipitates separated at once. The experiment was repeated without the addition of any acid; no brown colour appeared. The experiment was repeated, adding 5 cc. HCl (1·12); no brown precipitate separated in one hour, but the solution became blue and a blue precipitate separated slowly on account of the decomposition of the ferrocyanide. These experiments show that a little acid must be present, but that much acid makes the test less delicate.

0·1 and 0·2 mgrm. U as $UO_2(NO_3)_2$ were treated by *P. 58*d*, a few mgrms. PO_4 as Na_2HPO_4 being added; a good test was obtained with 0·2 mgrm., but only a very poor one after several minutes with 0·1 mgrm., showing that 0·1 mgrm. is about the limit of detectability, and that phosphate does not interfere with the detection of 0·2 mgrm. U.

The experiment with 0·5 mgrm. U and 0·5 cc. HCl (1·12) was repeated, except that water was added instead of NaCl solution; a dark red colour resulted, and no precipitate separated out in one hour, showing that NaCl is needed to coagulate the colloid.

(To be continued).

Royal Instititution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 7th inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, in the Chair. Dr. W. S. Anderson Griffith was elected a Member. His Serene Highness Albert Prince of Monaco, Membre de l'Institut, was elected an Honorary Member. Dr. F. Walker Mott, F.R.S., was elected Fullerian Professor of Physiology in the Royal Institution. The Special Thanks of the Members were returned to Dr. Ludwig Mond for a donation to the Fund for the Promotion of Experimental Research at Low Temperatures.

THE MONUMENT TO ROBERT BUNSEN.

By NICHOLAS KNIGHT, Cornell College, Mount Vernon, Iowa.

Some four or five years ago an appeal was sent out to the members of the German Chemical Society asking contributions for a suitable monument to the memory of Robert Bunsen. The monument was to be erected in Heidelberg, where the great teacher and investigator had laboured so successfully for thirty-seven years, from 1852 to 1889. The admirers of Bunsen throughout the civilised world generously responded, and with some assistance from the city of Heidelberg 70,000 marks were contributed for the memorial.

The monument was unveiled on August 1 of the present year. It consists of a life-size figure in bronze of the distinguished scientist, representing him as he appeared in 1859, about the time that he and Kirchhoff invented the spectroscope. In one hand he bears a manuscript, as if about to deliver a lecture. A flight of a few steps leads from the street to the monument. On each corner of the monument plot nearest the street is a colossal figure of white granite from the Black Forest, one representing the known sciences, and the other science awaking. The monument is not far from the Heidelberg Chemical Laboratory, a part of which was constructed under Bunsen's supervision, and was afterwards added to by his illustrious successor, the late Victor Meyer. The designer of the memorial is Professor H. Volz, of Karlsruhe, and from an artistic standpoint it is a meritorious work.

The exercises connected with the unveiling were simple but interesting and impressive. Music was furnished by the city orchestra, led by Professor Grau. Professor Theodore Curtius, the director of the Heidelberg Laboratory, the successor of Bunsen and Victor Meyer, as a member of the monument committee, gave an interesting detailed history of the enterprise. Professor Bernthsen, representing the industrial chemists, spoke at length of the great debt applied chemistry owes to the investigations of him to honour the memory of whom they were assembled. He spoke of Bunsen's contributions to industrial chemistry by the spectroscope, by his discovery of important technical analytical methods, by his work in volumetric analysis, contributing to the subject a new chapter on iodometry; by his studies in gas analysis; by his improved methods of iron production, and by his separation of various metals by electrolysis. Bernthsen was formerly a student and colleague of Bunsen, and he could speak with some authority. The mayor, Dr. Wilckens, accepted the monument in the name of the university city. Representatives of the German Chemical Society and a score of similar organisations deposited upon the monument laurel wreaths and other floral tributes suitably inscribed, as a mark of esteem for the man who so ably served his day and generation. Besides the scientific organisations, many of the German universities and technical schools sent their representatives and floral offerings to do honour to the occasion, which proved a notable day in the history of the city. The exercises were largely attended, and several members of Bunsen's family were interested spectators.

Bunsen was not only one of the foremost scientists of his day, but he was a good citizen and an amiable man, beloved by all classes. He received many honours in his lifetime. The name of the street on which he lived during the ten years that he was professor *emeritus* was changed to Bunsen Strasse—rather an unusual honour, but one richly deserved.

Bunsen came to Heidelberg as professor at the age of forty-one. During the thirteen years previous he had served the Universities of Breslau and Marburg, at both of which his work justified the expectations that were realised at Heidelberg.

Some of the visitors to the unveiling ceremonies wandered to the cemetery of the old university town on the Neckar, and sought out the grave of the great scientist. The monument is plain and simple, and bears a simple inscription. The mound is covered with rhododendrons. Not far away is the granite block, almost overgrown with ivy, which marks the last resting-place of that other world renowned scientist, Victor Meyer, the worthy successor of the immortal Bunsen.

NOTICES OF BOOKS.

Hints for Crystal Drawing. By Margaret Reeks. London, New York, Bombay, and Calcutta: Longmans, Green, and Co. 1908.

The difficulties with which the student of crystallography meets in crystal drawing are of considerable magnitude, and both teachers and pupils will welcome this lucid exposition of the simplest methods of dealing with the different systems. The author's solutions of the graphical problems are frequently most ingenious, and even the student whose knowledge of both mathematical drawing and crystallography is small will find that the easy stages by which he advances to the more complicated forms help him to overcome difficulties by degrees, and to acquire a thorough knowledge of each crystalline system. Clinographic projections are employed, and the clear illustrations of the most complex as well as the simpler forms make the text very easy to follow. Every student of mineralogy should have some knowledge of the elements of crystal drawing, and he will be able to find no better book from which to learn it; as a supplement to larger works on crystallography the book must be regarded as one of great value.

Einführung in die Kolloidchemie. ("Introduction to the Chemistry of Colloids"). By Dr. Viktor Pöschl. Dresden: Theodor Steinkopff. 1908.

This pamphlet gives a brief abstract of the most important developments of our knowledge of colloid chemistry, since the first colloidal substances were prepared by Graham, who, in 1861, laid the foundations of the subject. The nomenclature which has now been generally adopted is explained, and the peculiar properties of colloids are discussed as well as the preparation of the different groups of compounds. An interesting review is given of ultra-microscopic methods of research, and the fundamental principles of modern theories are very shortly expounded. The importance of a knowledge, even if only meagre, of the chemistry of colloidal substances is rapidly increasing, and its special relations to other sciences and to chemical industries are continually being extended. Every student of chemistry and every technical chemist should make time to study carefully some such work as this, which in the smallest possible space gives an adequate account of an interesting subject.

Synthetische und Isolierte Riechstoffe und deren Darstellung. ("Synthetic and Isolated Perfumes and their Preparation"). By Dr. Rudolf Knoll. Halle-a.-S.: Wilhelm Knapp. 1908.

The first and general part of this monograph gives accounts of the properties of perfumes, and discusses shortly the connection between chemical constitution and odour. This highly interesting subject, upon which important work has been done, is perhaps regarded as of a too purely scientific nature to come much within the scope of a book which is meant to be of special service to practical chemists, or to those who are just beginning their technical career. The account of general methods of synthetising perfumes includes descriptions of the preparation on a large scale of some substances, such as phosphorus trichloride, which are employed in making perfumes. The special part of the book deals with the constitution, properties, and preparation of compounds of aromatic odour, those belonging to the aldehyde and ketone classes being

grouped together. The methods of preparing them and the apparatus usually employed are described in outline, and in spite of the difficulty in getting information about processes which very often the manufacturers wish to keep to themselves as much as possible, the most representative methods on a technical scale are well described, as well as the purely laboratory processes of which they are modifications.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 18, November 2, 1908.

Separation of Tungstic Acid and Silica.—Paul Nicolardot.—Silica is not attacked even when combined with tungstic acid when it is treated with a mixture of imperfectly dried air and carbon tetrachloride vapour. Chloroform at 500° attacks tungstic acid, but has no action on silica; the vapours may be cooled in a condenser, and the method can be used to obtain tungstic acid free from silica.

Determination of the Atomic Weight of the Simple Ponderable Substance, Pantogen.—G. D. Hinrichs.—Hydrogen is the simplest of all the chemical elements, the least condensed form of ponderable material, and by the study of it the author has endeavoured to arrive at the atomic weight π of the fundamental substance pantogen. The atomic weight of hydrogen (O = 16) is very approximately $H = 16/16 - \frac{1}{8} = 1{\cdot}00787$. From theoretical considerations the author deduces that the excess over H = 1 of the true value of the atomic weight of hydrogen is the exact value of the atomic weight of pantogen for O = 16 or C = 12. The union of pantogen atoms in cubes, prisms, &c., gives the atoms of other elements such as nitrogen, oxygen, fluorine, and from the number of pantogen atoms forming the atom of the element its atomic weight may be calculated.

Zinc Phosphides.—Pierre Jolibois.—By pouring fused zinc on to red phosphorus and heating in a Perrot furnace crystals of a definite compound are obtained. Its formula is Zn_3P_2. With concentrated or dilute hydrochloric acid it yields PH_3 quantitatively according to the equation $Zn_3P_2 + 6HCl = 3ZnCl_2 + 2PH_3$. When phosphorus vapour acts on this compound *in vacuo* at 400° a brown mass is obtained, from which a black compound can be isolated. Its formula is ZnP_2. It dissociates at 500° into P_2Zn_3 and phosphorus. With hydrochloric acid it yields PH_3 and the solid hydride P_2H.

Influence of Neutral Salts on the Hydrolysis of Iron Perchloride.—G. Malfitano and L. Michel.—The hydrolysis of $FeCl_3$ in presence of KCl is either accelerated or retarded according to the relative proportions of the two salts. The composition and state of division of the colloidal matter are both affected, though the potassium chloride does not enter into the composition of the colloid. The other chlorides, NaCl, NH_4Cl, $BaCl_2$, $MgCl_2$, behave similarly, variations in their effect being due to their different coefficients of electrolytic dissociation. KNO_3 behaves like the chlorides. On the other hand, K_2SO_4 and K_2HPO_4, in which the negative radicle is polyvalent, act quite differently. They always hinder hydrolysis, and the lower the temperature the less they hinder it. In presence of increasing amounts of salts the number of iron ions is diminished, and the formation of $Fe(OH)_3$ is retarded if the proportion of salt added is not sufficient to diminish ionisation, and thus the antagonistic activity of the other product of hydrolysis, the acid.

Aloësol.—E. Léger.—When potassium chlorate is added to a solution of Cape or Uganda aloes in concentrated hydrochloric acid, a mixture of chlorinated aloïns is obtained, and a substance $C_{11}H_4Cl_4O_3$, which may be made to crystallise in almost colourless needles. One of the oxygen atoms of this compound belongs to a phenol OH, and the compound may be regarded as the tetrachlorinated derivative of an unknown phenol, to which the author has given the name aloësol. Tetrachloraloësol may be separated from the substances which accompany it by treating with hot alcohol, in which it is almost insoluble. The pink raw product may be purified by crystallising in boiling acetic acid after treating the solution with animal charcoal. Nitric acid converts it into oxalic acid and perchlorinated quinone, which shows that it contains a benzene nucleus in which two chlorine atoms and a hydroxyl group are substituted, and to which the residue $C_5H_2Cl_2O_2$ is attached. This residue contains neither alcohol, acid anhydride, ether-oxide, nor quinone functions.

MISCELLANEOUS.

Royal Institution.—The following are the Lecture Arrangements at the Royal Institution, before Easter:—Prof. W. Stirling, a Christmas Course of six experimentally illustrated lectures on "The Wheel of Life," adapted to a juvenile auditory; Prof. Karl Pearson, two lectures on "Albinism in Man"; Prof. A. A. Macdonnell, three lectures on "The Architectural and Sculptural Antiquities of India"; Dr. F. Walker Mott, six lectures on the "Evolution of the Brain as an Organ of Mind"; Prof. J. O. Arnold, two lectures on "Mysteries of Metals"; Mr. William Archer, two lectures on "The Revival of Modern Drama"; Dr. Hans Gadow, three lectures on "Problems of Geographical Distribution in Mexico"; Mr. A. D. Hall, two lectures on "Recent Advances in Agricultural Science"; Prof. G. H. Bryan, two lectures on "Aërial Flight in Theory and Practice"; Prof. Sir Hubert von Herkomer, two lectures on—(1) "The Critical Faculty," (2) "Sight and Seeing"; Sir Alexander Mackenzie, three lectures on—(1) "Mendelssohn" (with Musical Illustrations), (2, 3) "Chamber Music" (with the kind assistance of the members of the Hans Wessely Quartette); and Prof. Sir J. J. Thomson, six lectures on "Properties of Matter." The Friday Evening Meetings will commence on January 22, when Dr. Alfred Russel Wallace will deliver a Discourse on "The World of Life, as Visualised and Interpreted by Darwinism." Succeeding Discourses will probably be given by Lieut.-Colonel Sir Frederic Nathan, Prof. J. G. Frazer, Prof. H. A. Wilson, Sir Henry Cunynghame, The Right Hon. the Earl of Berkeley, The Right Hon. Viscount Esher, Mr. S. G. Brown, Mr. R. Threlfall, Mr. A. S. Eddington, Prof. Sir J. J. Thomson, and other gentlemen.

MEETINGS FOR THE WEEK.

MONDAY, 14th.—Royal Society of Arts, 8. (Cantor Lecture). "Twenty Years Progress in Explosives," by O. Guttmann.

TUESDAY, 15th.—Faraday Society, 8. "Re-determination of the Electrolytic Potentials of Silver and Thallium," by F. J. Brislee. "Heats of Combustion of Aluminium, Calcium, and Magnesium," by F. E. Weston and H. R. Ellis. "Formation of Graphite by the Interaction of Magnesium Powder and Carbonates," by H. R. Ellis. "Colloidal Barium Sulphate," by E. Feilmann.

WEDNESDAY, 16th.—Royal Society of Arts, 8. "London Milk Supply from a Farmer's Point of View," by Primrose McConnell.

—— Microscopical, 8. "Workshop Microscope for the Examination of Opaque Objects" and "A Simple Method of Illuminating Opaque Objects," by J. E. Stead. "Mounting Rotifers and Protista in Canada Balsam," by Rev. E. Tozer.

THURSDAY, 17th.—Chemical, 8.30. "Silicon Researches—XI., Silicotetrapyrrol; XII., Action of Silicon Chloroform on Potassium Pyrrol; XIII., Silicon Halides and Pyridine, Acetonitrile, &c.," by J. E. Reynolds. "Affinity Values of Tropine and its Derivatives," by V. H. Veley.

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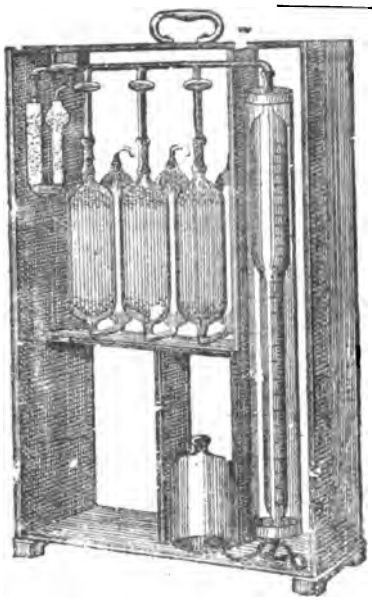

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ON SCANDIUM.*

By Sir WILLIAM CROOKES, D.Sc., F.R.S.

(Continued from p. 285).

Scandium Acetate, $(CH_3COO)_2ScOH,2H_2O$.

Scandium oxide after ignition does not dissolve in acetic acid. The hydroxide, however, dissolves easily, and the acetate soon separates from the liquid in the form of fine white crystals, difficultly soluble in water. The aqueous solution, evaporated over the water-bath, deposits hard crystalline crusts.

When freshly prepared, the acetate dried in the air has the composition given below. Water, however, is slowly given off, and after standing some days the salt passes into a monohydrate analogous to the formate.

When ignited, the acetate leaves a grey residue of scandia containing carbon. By ignition alone it is difficult to get it of a constant weight. The best way to analyse the salt is to moisten the weighed acetate with a few drops of nitric acid and cautiously to evaporate to dryness, heat, and ignite. In this way is obtained pure white scandia.

Analysis.

1. 7·795 grs. of acetate left 2·449 grs. of scandia, = 1·5859 grs. of scandium.
2. 7·133 grs. left 2·270 grs. of scandia, = 1·4697 grs. of scandium.
3. 19·035 grs. left on ignition 6·000 grs. of scandia, = 3·8848 grs. of scandium.

	Theory.		Experiment.		
			1.	2.	3.
Sc	44·100	20·49	20·35	20·60	20·41
$(CH_3COO)_2$ OH .. .. $2H_2O$ . ..	171·088	79·51	79·65	79·40	79·59
	215·188	100·00	100·00	100·00	100·00

After being exposed to dry air for several days, the dihydrate loses one molecule of water of crystallisation, and becomes a monohydrate—

$(CH_3COO)_2ScOH,H_2O$.

Analysis.

14·690 grs. left 5·262 grs. of scandia, = 3·4075 grs. of scandium.

	Theory.		Experiment.
Sc	44·100	22·37	23·19
$(CH_3COO)_2$ .. OH . .. .. H_2O	153·072	77·63	76·81
	197 172	100·00	100·00

Scandium Propionate, $(C_2H_5COO)_2ScOH$.

Propionic acid readily attacks scandium hydroxide, forming a difficultly soluble white amorphous salt. On evaporating the excess of acid the scandium propionate is left as a voluminous white powder. 100 parts of cold water dissolve 1·23 parts of scandium propionate, the greater part being precipitated on heating the solution, and re-dissolved on cooling. When boiled in water, the salt floats greasily about the surface. The propionate is easily soluble in alcohol even in the cold, and when the alcoholic solution is allowed to evaporate the propionate is deposited as a mass of minute spherical concretions with no appearance under the microscope of crystalline structure.

The scandium propionate so obtained has the formula—

$(C_2H_5COO)_2ScOH$.

Analysis.

15·978 grs. on ignition yielded 5·287 grs. of scandia, = 3·424 grs. of scandium.

	Theory.		Experiment.
Sc	44·100	21·29	21·43
OH .. .. .. $(C_2H_5COO)_2$ ..	163·088	78·71	78·57
	207·188	100·00	100·00

Scandium Butyrate, $(CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}COO)_2ScOH$.

When scandium hydroxide is heated with butyric acid, a white amorphous and difficultly soluble salt is produced. When boiled in water, the butyrate softens to a plastic mass, becoming hard and brittle on cooling. Like other scandium salts of the fatty acids, scandium butyrate is more soluble in cold than in hot water, and is precipitated from a cold solution on boiling. It is easily soluble in alcohol. The solution in cold water, evaporated over a water-bath, leaves a residue which under the microscope is seen to consist of minute spherical groups, radiating from a central nucleus.

Analysis.

6·251 grs. of butyrate yielded on ignition 1·826 grs. scandia, = 1·182 grs. of scandium.

	Theory.		Experiment.
Sc	44·100	18·75	18·91
OH .. .. .. 2(C_3H_7COO) ..	191·120	81·25	81·09
	235·220	100·00	100·00

Scandium Iso-butyrate, $\left(\begin{matrix}CH_3\\CH_3\end{matrix}\!>\!CH{\cdot}COO\right)_2ScOH,2H_2O$.

This salt is prepared in a similar manner to the butyrate, which it much resembles in appearance. It is more soluble in cold than in hot water, a saturated cold solution becoming thick and depositing amorphous flakes when heated. On cooling the liquid becomes clear again; in this respect it closely resembles calcium butyrate. It is very soluble in alcohol and is precipitated by water from the alcoholic solution.

The formula of the iso-butyrate differs from that of the butyrate in having two molecules of water of crystallisation :—

$(C_3H_7COO)_2ScOH,2H_2O$.

Analysis.

9·994 grs. of scandium iso-butyrate gave on ignition 2·550 grs. of scandia, = 1·651 grs. of scandium.

	Theory.		Experiment.
Sc_2	44·100	16·26	16·52
OH .. .. .. 2(C_3H_7COO) .. $2H_2O$	227·152	83·74	83·48
	271·252	100·00	100·00

* A Paper read before the Royal Society, April 30, 1908.

Scandium Iso-valerate,

$(C_4H_9COO)_2ScOH,2H_2O$.

Scandium hydroxide is readily attacked by aqueous iso-valeric acid, forming a white amorphous salt, soluble in cold water, precipitated on boiling, and again dissolved on cooling. Dried over sulphuric acid the iso-valerate has the composition—

$(C_4H_9COO)_2Sc(OH),2H_2O$.

Analysis.

10·902 grs. of scandium iso-valerate left on ignition 2·493 grs. of scandia, = 1·614 grs. of scandium.

	Theory.		Experiment.
Sc	44·100	14·74	14·80
OH, $2(C_4H_9COO)$, $2H_2O$	253·184	85·26	85·20
	299·284	100·00	100·00

Scandium Oxalate,

$Sc_2(C_2O_4)_3,5H_2O$.

Scandium oxalate comes down as a white crystalline powder when oxalic acid or ammonium oxalate is added to a solution of scandium. If much free mineral acid is present the precipitation is not immediate; it begins by a cloudiness, which in a few minutes increases to a crystalline precipitate, a little scandium oxalate remaining in the solution. It is slightly soluble in water, weak acids, and more so in solution of ammonium oxalate. On boiling a mixed oxalate from wiikite in a solution of ammonium oxalate the portion dissolved was richer in scandium than the insoluble part. Attempts were made to effect a separation of scandia from the accompanying earths by making use of this fact. A mixed oxalate of crude earths was boiled in a strong solution of ammonium oxalate and then filtered, and this operation was repeated on the residue several times. Ultimately the most soluble and the least soluble earths were separated and their spectra photographed. The most soluble earth was nearly all scandia, with a little yttria, and traces of ytterbia, titania, and lime. The least soluble contained more yttria than scandia, but not much of either. By a repetition of this process it would not be difficult to effect a good separation of scandia and yttria; I think the fused nitrate process is simpler and quicker.

When crude scandium oxalate is boiled in a solution of ammonium oxalate a partial separation of the earths is effected, but it is less complete than when ammonium carbonate is used as a solvent. This is a very good method for separating scandia from zirconia, as hardly a trace of the zirconia is to be seen in the portion insoluble in hot ammonium oxalate.

According to Nilson (*Comptes Rendus*, vol. xci., p. 121), the precipitated oxalate is slightly soluble in weak acids, and even in water. I have verified this observation; care must be taken not to over-wash the oxalate, or loss will ensue, and the filtrates should be kept and worked again for scandium.

The precipitated oxalate, dried in the air, contains 5 molecules of water.

Analysis.

1. 22·445 grs., ignited at a yellow heat, left 6·988 grs. of scandia.
2. 21·873 grs. ignited left 6·806 grs. of scandia.

	Theory.		Experiment.	
			1.	2.
Sc_2O_3	136·20	30·79	31·13	31·12
$3C_2O_3$, $5H_2O$	306·08	69·21	68·87	68·88
	442·28	100·00	100·00	100·00

When the 5-hydrated oxalate is allowed to dry at the ordinary temperature over sulphuric acid, it loses 2 molecules of water and becomes a 3-hydrate—

$Sc_2(C_2O_4)_3,3H_2O$.

Analysis.

7·639 grs. left on ignition at a yellow heat 2·529 grs. of scandia.

	Theory.		Experiment.
Sc_2O_3	136·200	35·33	33·11
$3C_2O_3$, $3H_2O$	270·048	66·47	66·89
	406·248	100·00	100·00

The 5-hydrate of scandium oxalate, dried in a hot-air oven at 100°, loses three molecules of water, and becomes a 2-hydrate—

$Sc_2(C_2O_4)_3,2H_2O$.

Analysis.

22·445 grs. lost 2·014 grs. of water.

	Theory.		Experiment.
Sc_2O_3, $3C_2O_3$	352·200	90·72	91·03
$2H_2O$	36·032	9·28	8·97
	388·232	100·00	100·00

The hydrated oxalate, dried *in vacuo* at 100° or in air at 140°, loses all but one molecule of water and becomes a mono-hydrate—

$Sc_2(C_2O_4)_3,H_2O$.

Analysis.

1. 32·118 grs. left on ignition 11·756 grs. of scandia.
2. 15·694 grs. left 5·815 grs. of scandia.
3. 8·304 grs. left 3·010 grs of scandia.

	Theory.		Experiment.		
			1.	2.	3.
Sc_2O_3	136·200	36·79	36·60	37·05	36·25
$3C_2O_3$, H_2O	234·016	63·21	63·40	62·95	63·75
	370·216	100·00	100·00	100·00	100·00

Scandium Succinate,

$$\begin{matrix} CH_2COOSc(OH)_2 \\ | \\ CH_2COOSc(OH)_2 \end{matrix} \quad H_2O = C_4H_8O_8Sc_2,H_2O.$$

This salt comes down as an insoluble white precipitate when dilute solutions of ammonium succinate and scandium nitrate are mixed. It also may be prepared by boiling scandium hydroxide in solution of succinic acid. It is slightly soluble in excess of either reagent, and is insoluble in water or alcohol. When dried in the air it forms a hard porcelain-like mass.

Analysis.

12·713 grs. of scandium succinate heated for ten hours to 180° lost 0·822 gr. of water, and when ignited to full redness it left 6·089 grs. of scandia, = 3·9431 grs. of scandium.

	Theory.			Experiment.
Sc_2	88·200		30·38	31·02
C_4	48·000			
H_8	8·064	184·064	63·41	62·51
O_8	128·000			
H_2O	18·016		6·21	6·47
	290·280		100·00	100·00

SCANDIUM PICRATE,
$\{C_6H_2(NO_2)_3O\}_2ScOH,14H_2O$.

The hydroxide dissolves readily in warm solution of picric acid and separates in groups of long needle-shaped crystals of a rich yellow colour. When rapidly crystallised from a strong solution, the crystals form confused feathery masses, which on pouring off the mother-liquor turn to a felt-like mass difficult to drain. 100 parts of water at 20° dissolve 0·95 part of scandium picrate. At 9° the same quantity of water dissolves 0·37 part. Dried by exposure to cold dry air the crystals have the composition shown above.

The picrate cannot be detonated when wrapped in tin-foil and struck on an anvil with a heavy hammer. Heated in a dry test-tube it darkens, gives a slight sublimate, fuses, and at a little higher temperature faintly explodes, leaving a black flocculent residue, which burns off to pure white scandia at a red heat.

The explosion, although not violent, is sufficient to blow some of the products out of the crucible. The scandia therefore was estimated by precipitating the hot solution with ammonia in considerable excess and well boiling the liquid before filtering. Unless this precaution is taken some of the picrate precipitates as such, and slight explosions occur when the precipitate is burnt.

Analysis.

1. 27·497 grs. of picrate, dried at 100°, gave off 5·906 grs. of water.
2. 27·497 grs. yielded 2·422 grs. of scandia, = 1·569 grs. of scandium.

	Theory.		Experiment.
			1 and 2.
Sc	44·100	5·73	5·71
OH, $2\{C_6H_2(NO_2)_3O\}$, $5H_2O$	563·180	73·20	72·81
$9H_2O$	162·144	21·07	21·48
	769·424	100·00	100·00

When the 14-hydrate is heated to 100°, nine molecules of water go off, leaving a 5-hydrate.

$\{C_6H_2(NO_2)_3O\}_2,ScOH,5H_2O$.

Analysis.

21·657 grs. of the picrate, dried at 100°, gave 2·422 grs. of scandia, = 1·569 grs. of scandium.

	Theory.		Experiment.
Sc	44·100	7·26	7·24
OH, $2\{C_6H_2(NO_2)_3O\}$, $5H_2O$	563·180	92·74	92·76
	607·280	100·00	100·00

SCANDIUM BENZOATE,
$(C_6H_5COO)_3Sc$.

Scandium benzoate comes down as a white curdy precipitate on adding a solution of ammonium benzoate to scandium nitrate solution. On boiling the solution the precipitate collects as a crystalline powder. It is slightly soluble in water and alcohol, and easily soluble in dilute mineral acids.

Analysis.

12·303 grs. of scandium benzoate left on ignition 2·204 grs. of scandia, = 1·4272 grs. of scandium.

	Theory.		Experiment.
Sc	44·10	10·83	11·60
C_{21}, H_{15}, O_6	252·00, 15·12, 96·00	89·17	88·40
	407·22	100·00	100·00

(To be continued).

THE DECOMPOSITION OF YTTERBIUM INTO ITS ELEMENTS.*

By C. AUER VON WELSBACH.

(Concluded from p. 225).

The Spark Spectra.

To produce the luminous vapour I used exclusively my new spark apparatus which depends upon the application of the spark at break, and which when it is properly managed and a rotating platinum pole is used, easily allows the spectra of most elements to be obtained almost pure and free from the lines of the pole substance as well as from those of air.

As regards their character, the spectra with the spark at break are in many respects intermediate between the arc spectra and the peculiar spark spectra. They are sometimes more like the former and sometimes resemble the latter, and at any rate with the arrangement I used, this variation in character does not seem to occur according to any fixed rule.

Thus, for example, the spark spectrum of platinum is almost identical with the arc spectrum, while the spectra with the spark at break of the rare earths agree perfectly with the spark spectrum, as Exner and Haschek's measurements show.

The lines of the arc spectrum of platinum coincide exactly with the lines of the spark at break spectrum. In no case could the slightest difference be detected.

The spark spectra of the two new elements are relatively poor in lines, but they belong to the brightest known. This holds good especially for cassiopeïum. The few lines which this substance has in the optical part of the spectrum somewhat recall the magnificent barium spectrum. Besides these lines the Cp spectrum is characterised by two channelled bright shining bands in the green and blue, somewhat recalling the Cy-spectrum. While the band in the green consists of several fine lines that in the blue is of a nebulous character. With the dispersion which my apparatus possesses the resolution into lines could not be effected. The light maxima lie towards the refrangible part of the spectrum; thus the structure is the opposite of that of the Cy bands.

The spectrum of Ad is much richer in lines than that of Cp, both in the optical and ultra-violet parts. Most of the Yb lines occur in it. To produce the spectrum a Rowland's concave grating of the old kind was used for all the photographs taken for reproduction or measurement. It possesses 14,473 lines per English inch, and a radius of curvature of about 2 m. The length of the divided surface amounts to about 54 mm. I prefer this grating, with which I have taken all comparison photographs of late years, to others, because, besides excellent definition, it possesses the property so valuable for my spectral work of not distributing the diffracted light uniformly, but concentrating it in certain spectral regions which are well situated for taking the photographs (*cf.*, H. Kayser, "Handbuch der Spektroskopie," vol. i., p. 433). For this reason I often succeeded in taking spectrographs of brilliant sharpness for the whole length of the spectrum, using only a few milligrammes of the substance under investigation.

If a sunbeam is allowed to fall on this grating in the direction of the optical axis, and the diffracted light is reflected towards a wall, the spectrum of the first order appears bright on one side, while that on the other side is quite faint, and the same holds good for the spectra of the next higher orders. Curiously enough the spectrum of the first order breaks off suddenly at about $\lambda 3300$, but occurs again in the second order of the same side with such great intensity that the photographs for the first and second order could be taken simultaneously with almost the same effect. In consequence for the spectral region from 3300 towards the shorter wave-lengths I have always taken photographs in the second order.

* *Monatshefte für Chemie*, February, 1908.

The result of the great sensitiveness of our ordinary dry plates towards light of a short wave-length is that the zone within which lines of the first and second order can be taken together is fairly large, comprising about 800 A.U.

Since in many experiments, as for instance in testing very expensive substances, it is of great importance to obtain a spectral region comprising several thousand A.U. in one single photograph suitable for measuring or comparison, I used a concave mirror silvered on the inner surface to collect the light instead of the usual quartz lens.

All waves of 235 $\mu\mu$ and upwards were not reflected to an appreciable extent by the mirror, as silver is transparent to such rays, so that λ4600 of the first order could be obtained free from lines of the second order. The part of the spectrum suitable for measurement taken in one photograph on ordinary bromine plates reaches from λ4600 to 2500. The use of concave mirrors is to be recommended on account of the great strength of the light which the spectra receive, especially if the parts of the visible spectrum which do not act on the ordinary plates are to be taken. Moreover, this is of great importance for many comparative chemical investigations apart from wave-length measurements. For it is in this part of the spectrum that differences in the intensity of the lines of two spectra produced by alterations in the mixture are to be detected long before they can be recognised in the ultra-violet part.

Grating, slit, as well as the boxes for the cassettes made of flexible material, are placed at fixed positions on a strong frame made of perfectly dry mahogany. The whole apparatus is enclosed in a roomy box made of mahogany and covered on the interior with dull black velvet. The frame, lying freely on rubber balls, rests on an iron stand. The whole apparatus can be moved in any direction, and it is not more difficult to transport than another larger spectral apparatus. I give a fuller description of the whole arrangement, which presents many novelties, in my book, "Spektroskopische Methoden der Analytischen Chemie," which will shortly be published.

Almost all photographs were taken on very sensitive dry plates of the finest grain, which were about 0·8 mm. thick and thus flexible. As such thin glass plates can be cut very sharply after a little practice with a suitable diamond, without splitting the edge, it is easy to place two or more spectra taken on the plates very near to one another, so that they can conveniently be compared directly with one another, provided that they were photographed under exactly the same conditions.

Owing to the fixed position of grating, slit, and plate only a certain region of the spectrum was normal.

I did not ascribe any great importance to this because I did not originally intend to measure whole spectra by determining accurate wave-lengths, but I was rather aiming at getting the spectra in forms in which they could be directly compared with one another. For all those cases in which lines have to be measured, or in which it is a question of determining accurately the position of a line, I wished to obtain the desired information within a few minutes without great trouble and without elaborate apparatus. For this reason I abandoned the usual classical comparison methods and measured the spectra by photographed scales corresponding almost exactly to the length of the spectrogram. But the lack of sharpness of the divisions on the printed scales of course diminished the accuracy of the measurements, very often more than was expected, and also strained the eye very considerably.

I succeeded later in avoiding this error, and photographed the scale so sharply that it allowed of a considerable enlargement without specially straining the eyes. This method was suited for taking fairly accurate measurements; thus in the spectrum of the second order the A.U. corresponded to about 0·27 mm. of the photograph. I used the platinum lines as standard lines. I took the wave-lengths of these from H. Kayser's tables of the arc spectrum of platinum. I rounded off the numbers, which give the wave-lengths to thousandths of an A.U., to two places of decimals for my purpose.

The differences which exist between the wave-lengths of Kayser (arc spectrum) and those of Exner and Haschek (spark spectrum) mostly account for the discrepancies which appear between my measurements and those of the latter investigators.

As the number of platinum lines in the optical part is too small to make it possible to take the necessary measurements I used the lines of the platinum spectrum of the second order, which I took between the two other spectra, much shortened and thus made easily recognisable. The platinum lines of the first order contained in the spectrum of the substance to be tested thus provided a control of any displacement.

I could not prove any deflection of the lines in consequence of the high altitude of the place at which the photographs were taken (800 m. above the sea level).

The breadth of the slit was always about 0·01 mm.

As source of current a storage battery of terminal voltage 50 volts, built for lighting supply, was used. In all the photographs described in this article a constant resistance of 25 ohms was placed in the circuit. The intensity available to produce the spark was therefore fairly small, so that the positive poles made of platinum wire 1·4 mm. thick only glowed faintly at their ends.

I used ethyl red as sensitiser for the photographs in the visible part. As various accounts have been published of the sensitising action of this dye I may regard it as well known. On account of their high sensitiveness I have mostly worked with wet plates, and I always exposed them immediately after sensitising, because if they lie by, on account of the carbon dioxide contained in the air, the sensitiveness rapidly decreases. I brushed off adhering drops of water by means of a broad fine brush. It is scarcely necessary to point out that the gradation of the sensitised plates leaves much to be desired.

Partly that I might better estimate the intensity of the lines, and partly in order to recognise for certain any impurities present, I took several photographs with different times of exposure under as far as possible the same conditions and for the same region of the spectrum. Thus for the region from λ4600 to 2500 I used exposures of 1, 10, 100, 180, and 200 seconds, and with Cp 500 seconds so as to find any trace of impurity present. In the visible part I was satisfied with three photographs with exposures of 60, 200, and 600 seconds. The estimation of the intensity of the lines in this part I also performed occasionally by ocular observation, because the sensitised plates possess limits of sensibility quite different from those of ordinary plates and much more irregular. Naturally these, like other numbers, are devoid of scientific foundation.

In a table in the original paper I have collected the results of the measurements obtained with the help of the above named plates, in so far as they refer to the more intense lines.

In the region 4600 to 2500 all strong lines are marked which were to be seen on the plates after an illumination of 100 seconds, excluding those which came from the substance of the poles, such as platinum lines and some lines of Pd, Ir, and Cu.

I have chosen this particular time of illumination because these photographs were very much like those of Exner and Haschek, as far as the intensity of the lines is concerned, and thus the statements of these investigators could be compared with mine. For completeness sake I have also taken those wave-lengths which Exner and Haschek give for the arc spectrum of ytterbium. The spectral work of Exner and Haschek, which is of such importance in chemical investigation, I assume to be known.

From the relative intensity of the lines for the region 4600 to 2500 it is seen that the spectrum with the spark at break of the rare earths in question is very similar to the spark spectrum produced by high tensioned currents.

For all measurements made in the region of the first order, *i.e.*, from 4600 to 3300, I have given Exner's numbers generally unaltered when those obtained by me did not differ from them by more than 0·05 A.U. For the measure

ments in the reign of the second order I have lowered the difference to about 0·03 A.U. In this region there are consequently many numbers which differ from those of Exner. These differences, however, in most cases are to be ascribed to the differences already mentioned as existing between Kayser's and Exner's numbers.

To supplement the table I have added a photographic representation of the spectra. Although the difficulties of getting a good reproduction of a spectral photograph are very great, they do not nevertheless seem to me to be worth taking into account in comparison with the advantages which a true picture offers. For, firstly, the spark spectra of the new elements in relation to one another and also to ytterbium—and this is much the most important in this work—can be described shortly and intelligibly only by the help of good pictures; secondly, we are dealing with rare elements difficult to prepare, which even in the small quantities necessary to obtain a spark are either obtained only with great difficulty or not at all, although their spectra, and this holds good especially for Cp, are characterised not only by the position of the lines but also by their constitution and character.

With good reproductions with the help of wave-length tables we can easily take photographs directly comparable for each instrument, photographs by means of which a spectrum to be investigated can be quickly and easily tested. This is all that is of importance in many cases of chemical investigations. It is of course supposed that to produce the luminous vapour giving the spectrum the same arrangements are made under the same experimental conditions. All pictures were obtained directly by the photographic method. They include the region from 6000 to 2600.

To develope the plates I used only hydroquinone. In order to get the half tones as well as the fainter lines, according to the advice of practical photographers I avoided everything which could appreciably increase the contrasts. The pictures have thus undoubtedly lost much as engravings, but have gained considerably in clearness.

To produce the spark I used only pure aqueous nitrate solutions containing about 10 per cent of oxide.

The general examination of the figures shows the great difference between the spectra of Ad and Cp in the optical part, the region photographed on the sensitised plates. Here the spectrum of ytterbium, except of course in so far as the intensity of the lines is concerned, plainly divides itself into the two others. This is not quite the case in the violet and ultra-violet part, the special region of our highly sensitive bromine plates. Although the ytterbium spectrum in these regions also appears fairly clearly as the sum of the two other spectra, these latter are much more like one another. For almost every strong line in the spectrum of one element has a corresponding line visible, if faint, in the spectrum of the other. I have previously alluded to this peculiar phenomenon, which is important in the application of spectral analysis in analytical chemistry.

In the given case the objection might be raised that because the time of illumination was the same for all plates, the violet and ultra-violet rays, in consequence of the high sensitiveness to light of the plates, would act more strongly than those situated in the visible part. This may hold good for the yellow rays, but it is not true for the blue-green. The true causes are much more obscure.

The differences mentioned appear still more clearly when the platinum lines are considered. As only platinum poles were used to produce the spark, all substances brought on to the poles during the production of the spark must to a certain extent be adulterated with platinum, or rather the substance giving the spark consists of an alloy of earth metal and platinum such as can actually be shown to exist chemically in the peripheral part of the pole. Suppose the behaviour of the line λ 5301·18 in the blue-green is compared with that of the line λ 2998·09 in the ultra-violet. While the one, in spite of its extraordinary intensity, can hardly be detected in the spectrum of the earth, the other, although much less strong, is sharp and clearly visible in all spectra. In order to make this marked difference still plainer I magnified the spectral zone from 4700 to 4530 to twice its size after taking it on the sensitised plate, and also after taking it on the ordinary bromide plate. As regards the latter I must mention that it was done intentionally without a ray filter because in it some faint, but not disturbing, lines of the second order are found. The platinum line 4552·59 is observed in both parts.

In the platinum spectrum of the sensitised plate this line is very intense, but, on the other hand, it is scarcely visible in the earth spectrum; while in the platinum spectrum on the bromide plate it is not strong, it appears bright and sharp in the earth spectrum.

If now the photographs of the sensitised plates are compared ocularly with the spectrum itself a striking agreement is observed on the whole, a phenomenon which allows of a very simple explanation. What holds good for the lines described also holds good for all other lines concerned, and, as far as I can see at present, for all other spectra. The sensitiveness of the spectral reaction in the violet and ultra-violet part of the spectrum (the active region of the ordinary dry plate) is far greater than in the visible part and steadily increases in this part towards the less refrangible end. Hence in all cases in which incipient variations in mixtures are to be established the spectral tests must be performed in the visible part, and either ocularly or with wet plates as highly sensitised as possible. I have alluded to these relations very shortly here, partly because they seem to me to be important for the right estimation of the tables, and partly because I believe that the sooner they are taken into consideration the better after the previous neglect of them in chemical work.

An exhaustive treatment of them I must leave for a later article, all the more because this would not be possible without a thorough discussion of the nature of the photo-chemical processes which occur in the plate, and which still need much explanation.

In all spectra several Ca lines occur, especially λ3968·16 and 3933·80. The intensity of the first line in the wave-length tables of Exner and Haschek was denoted by 500, and that of the latter by 1000. Small traces of calcium could be detected in all preparations. It was not possible to remove it completely from the earths by the usual methods of analytical chemistry. Other methods which would have led to complete separation could not be applied owing to the small quantity of the specimens obtainable.

The constant appearance of calcium in these fractions, which it will be remembered had been obtained by fractional crystallisation from ammoniacal ammonium oxalate solutions, was very surprising. I therefore tested for calcium in these preparations. By careful precipitation of the well boiled earth metal solutions containing excess of ammonium salt with ammonia free from CO_2 I succeeded in getting a minute amount of pure calcium. The spark spectrum given by this specimen was identical with the normal spectrum of calcium; no single line showed any difference in intensity.

In the ultra-violet part of the Ad spectrum lines are to be seen which are either missing or nearly so in the ytterbium spectrum. They are the lines of elementary thulium.

Finally, I must mention that in the Cp spectrum, when the time of exposure amounted to 500 seconds, very faint iron lines and a very faint Mg line could be detected. There were no lines of other substances.

Several lines, especially in the ultra-violet, occur in all three spectra with the same intensity. I have marked them *g. l.* in the table, *i.e.*, common lines.

It is not probable that these are only apparent coincidences. For this peculiar phenomenon appears with all elements which are closely allied chemically, *e.g.*, neodymium and praseodymium.

Finally, I have still to mention a third group of lines which are strongest in the spectra of the middle fractions; they appear specially clearly on the sensitised plates after a long time of illumination (600 seconds).

To these belong the lines 5104·60, 5067·40, 5009·72, which in the spectra of Ad and Cp show no corresponding marked lines. All these lines indicate that between Ad and Cp a third Yb element exists if in small quantities only. Unfortunately it is hopeless to try to get this element, even in a half pure state. For the separation processes, even supposing that the tendency to separate remained the same, which is not certain, would take many centuries.

To protect myself against claims of priority which have recently been made, I must make a short personal explanation. I proved that ytterbium could be decomposed into two new elements at the beginning of 1905.

In March of the same year I reported this discovery to the Kaiserl. Akademie der Wissenschaften of Vienna. This short but quite positive statement appeared in the academic *Gazette*, No. x., 1905. The following sentences occur in it:—

"It (Yb) consists of two new elements. The spark spectra of the new elements are parts of the spectrum of ytterbium, and this may be regarded as the sum of the two new spectra. Some lines belong to the elements in common."

This report appeared afterwards in many technical papers, and so became widely known.

THE PRESENT STATE OF OUR KNOWLEDGE OF RADIO-ACTIVITY.*

By A. DEBIERNE.

(Concluded from p. 291).

IV.

Radio-active substances were discovered in certain special minerals, and the first researches showed that only minerals containing thorium or uranium are appreciably active. Since these researches extremely sensitive apparatus has been employed, and the presence of active substances has been revealed in a very large number of ordinary materials.

One of the most interesting results was obtained with the mineral waters and the mud deposited by medicinal springs. Extensive experiments by Pierre Curie and Laborde have shown that most mineral waters contain either small quantities of radium or else radium emanation. With some waters the proportion of emanation is fairly large, and it is thought that the curative properties may be due to the presence of the emanation. The researches of Moureu have shown the presence in these waters of helium, neon, and argon which always accompany the emanation.

It has also been proved that the emanation is present in measurable quantities in the atmosphere and in sea-water. Strutt has proved the presence of radium in most minerals, and has performed quantitative experiments, thus determining approximately the amount of radium present on the earth's surface. The result obtained has led to interesting conclusions.

The experiments of Pierre Curie having shown that radio-active transformations are important sources of energy, attempts have been made to find out if the production of energy in the universe may be due to the presence of active substances. It has been found that all the energy emitted by a star such as the sun may be explained by the presence of a relatively small quantity of radium, about 1 grm. per cubic metre.

Since Strutt's determinations enable us to form an approximate idea of the amount of radium on the surface of the earth, we can find out whether the corresponding liberation of heat is appreciable. The following is the result obtained:—

It is known that from a certain depth below the surface of the earth the temperature increases regularly, and determinations have been made of what is called the temperature gradient, *i.e.*, the variation of temperature per unit of length. The existence of a corresponding fall of temperature from the interior of the earth's crust to the exterior results in a continual transference of heat, which can be calculated if the temperature gradient and the mean conductivity for heat of the earth's crust are known. This heat is continually supplied by the interior of the globe, and it must be admitted that the earth is gradually cooling. But not only might the liberation of heat resulting from the presence of radium compensate this loss of heat, but also if it is supposed that the whole mass of the earth contains the same proportion of radium the temperature ought to rise instead of fall. This rise of temperature would be very slow and quite imperceptible.

The study of the phenomena of radio-activity may lead to results of importance in other geological problems. Thus, if we have an accurate knowledge of the mean duration of life of elements such as uranium or thorium, we can form an idea of the age of the minerals which contain them. The age of the minerals might also be calculated approximately by another method—the determination of the quantity of helium they contain, supposing this helium has remained imprisoned in the material. As the law of the production of helium is known approximately the age of the mineral can be deduced. The numbers thus obtained for the radio-active minerals (some hundreds of millions of years) are of the same order of magnitude as those which have been obtained by geologists by other lines of reasoning.

Researches have also been carried out to determine whether the property of radio-activity is limited to certain elements known to us, or whether it is a general property of matter. Such a result is by no means improbable, for we know substances which have very different activities; for instance, radium is several million times more active than uranium, and it is evident that if some substances are only one million times less active than uranium our methods of research would not enable us to detect them. The researches carried out in this direction have shown that space is traversed by a very feeble penetrating radiation, and also that certain ordinary substances appear to possess a true radio-activity. Lead and potassium give decidedly positive results, but it is difficult to decide whether the radio-activity detected is due to the elements themselves or to radio-active impurities.

V.

I will conclude this article by drawing attention to one very important aspect of radio-activity—the regularity and absolute constancy of the laws of the variation of radio-active transformations. It is true that these laws are identical with those which have been deduced for analogous chemical transformations, but they seem absolutely independent of the external conditions. This may be due to the atomic nature of the transformations; however, it is somewhat difficult to form a conception of the mechanism of the phenomenon.

In molecular chemical transformations the progressive transformation of the matter present may be explained by the fact that all the molecules are not in the same state, either owing to collisions with neighbouring molecules or to movements in the interior of the molecule; a molecule does not undergo the transformation in question, which is instantaneous, until, in consequence of these different movements, it reaches a certain state; the progressive law of transformation is a kind of law of chance, which is regular as long as the number of molecules is large and the external conditions remain the same; the same proportion of molecules reaches the state in question during a given interval of time and undergoes the chemical transformation. With radio-active transformations experience shows that external conditions of temperature, &c., have no influence, not even the state of combination; the collisions between the atoms can thus have no influence upon their particular state from the radio-active point of

* *Revue Générale des Sciences*, 1908., xix. 730.

view. The sole remaining possible influence which can intervene are the movements in the interior of the atom. But let us consider a certain quantity of radium C, for example; it may be supposed that at its moment of transformation each atom passes through the same state; this is in agreement with the fact that when an α particle is projected each particle is emitted with the same initial velocity. At the end of three hours almost all the radium C is transformed into radium D. Amongst all the atoms of radium D which start in the same state, at practically the same time, and which, moreover, are not influenced by external agencies—pressure, temperature, state of combination, &c.—some are immediately transformed into radium E, while others may wait for several hundred years before being transformed. In this conception of radio-active transformations there are some points which are difficult of comprehension. Perhaps a better explanation would be reached if it were supposed that the transformation is not absolutely spontaneous, and that an external cause exists for the evolution of the elements, but it is difficult to imagine such a cause.

Finally, it is not impossible that external conditions have a slight influence over the laws of evolution; the experiments which have been performed have up to the present given a negative result, but they have not been numerous, and it cannot be affirmed that we shall not some day succeed in exercising an influence over the production of radio-active transformations.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

Part III.—Analysis of the Aluminium and Iron Groups, including Beryllium, Uranium, Vanadium, Titanium, Zirconium, and Thallium.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Continued from p. 292).

Confirmatory Experiments and References (continued).

Behaviour of Vanadyl Salts and Vanadic Acid towards Potassium Ferrocyanide.—5 mgrms. V as Na_3VO_4 after evaporation with HCl (whereby it is reduced to vanadyl chloride) were treated by *P. 58*d*; a greenish yellow precipitate separated. The precipitate was filtered off, and the filtrate tested for vanadium by *P. 58*e*; only a very faint pink colour resulted, showing that the precipitation of the vanadium by the ferrocyanide is nearly complete. The experiment was repeated except that only two drops of HCl were added and the solution was not evaporated; no precipitate separated in half an hour, showing that a small quantity of vanadium in the form of vanadic acid is not precipitated by $K_4Fe(CN)_6$.

*P. 58*e*: *Non-interference of Lead with the Vanadium Test.*—A solution containing 2 mgrms. Pb as $Pb(NO_3)_2$, and 0·5 mgrm. V as Na_3VO_4 and 2 grms. NH_4NO_3 was treated by P. 58*e*, and the black precipitate of PbS formed was filtered off; on saturating completely with H_2S, the filtrate had the characteristic violet-red colour due to vanadium.

*P. 58*g*, N. 1: *Separation of Aluminium and Beryllium.*—See Havens (*Zeit. Anorg. Chem.*, 1898, xvi., 15).

Precipitation of Aluminium as $AlCl_3.6H_2O$.—0·5 mgrm. Al as $AlCl_3$ was treated by *P. 58*g*, the total volume of the acid ether mixture being about 15 cc.; on saturating with HCl gas no precipitate could be seen, but on standing about fifteen minutes a distinct crystalline precipitate separated out.

100 and 500 mgrms. Al as $AlCl_3$ were treated by *P. 58*g*; the filtrates, which had a total volume of about 70 cc., were evaporated to about 20 cc. and made alkaline with NH_4OH; no precipitate separated, but in the second experiment there was a faint turbidity corresponding perhaps to 0·1 mgrm. Al.

Behaviour of Beryllium, Uranium, and Chromium in the Hydrochloric Acid Ether Process.—100 mgrms. Be as $BeCl_2$ were treated by *P. 58*g*, the final volume being about 30 cc.; a small precipitate remained. This was filtered off, washed twice, dissolved in a little water, and proved to contain aluminium but no beryllium by boiling in a 10 per cent $NaHCO_3$ solution as described in *P. 58*h*.

500 mgrms. Al and 2 mgrms. Be as chlorides were treated by *P. 58*g* in a volume of 60 cc., and the filtrate was treated by *P. 58*h*; a good test for beryllium was obtained, and the amount of beryllium in the filtrate was estimated to be 1 or 2 mgrms., showing that very little beryllium was retained by the $AlCl_3.6H_2O$.

100 mgrms. U as UO_2Cl_2 were treated by *P. 58*g*, the total volume being 50 cc.; a clear yellow solution was obtained.

20 mgrms. Cr as K_2CrO_4 were boiled with HCl (1·20) to reduce the chromium to the chromic state, and the green solution was treated by *P. 58*g*, in a volume of about 30 cc.; the green colour quickly disappeared and a violet precipitate separated. This was filtered off after several hours and the filtrate tested for chromium by evaporating, adding NH_4OH, and boiling; a precipitate estimated to contain .5—10 mgrms. Cr separated.

*P. 58*h*, N. 1: *Separation of Beryllium and Aluminium in Strong Sodium Hydrogen Carbonate Solution.*—See Parsons and Barnes (*Journ. Am. Chem. Soc.*, 1906, xxviii., 1589).

200 mgrms. Al as nitrate were dissolved in 20 cc. water, and added to a warm solution of 10 grms. $NaHCO_3$ in 80 cc. water; the mixture was heated to boiling in a flask, boiled for one minute, and filtered; the filtrate was acidified with HNO_3, evaporated to about 20 cc., and made alkaline with NH_4OH; no precipitate separated on warming gently, nor on standing.

100 mgrms. Al and 1 mgrm. Be as nitrates were dissolved in 50 cc. water and 6 grms. $NaHCO_3$ were added; the mixture was heated to boiling, boiled for one minute, and filtered while still hot; the filtrate was made acid with HNO_3, evaporated to about 10 cc., and made alkaline with NH_4OH; a very small precipitate separated, which was estimated to contain 1/20 mgrm. Be. The experiment was repeated with 2 mgrms. Be; the result was the same. The experiment was repeated with 5 mgrms. Be; scarcely 1 mgrm. Be was found in the filtrate. Therefore small amounts of beryllium cannot be completely separated from 100 mgrms. Al by this method.

The precipitate obtained in the experiment with 1 mgrm. Be was treated by *P. 58*g—h*; the beryllium present was estimated to be nearly 1 mgrm., showing that this separation with HCl and ether is more satisfactory than that with 10 to 12 per cent $NaHCO_3$.

Behaviour of Iron in Strong Sodium Hyarogen Carbonate Solution, and its Precipitation as Sulphide.—2 and 5 mgrms. Fe as $FeCl_3$ were treated by the last paragraph of *P. 58*h*; small precipitates of $Fe(OH)_3$ were formed in the $NaHCO_3$ solutions, but the precipitation was incomplete, for after acidifying the filtrates and adding NH_4OH, precipitates of $Fe(OH)_3$ were obtained, each of which was estimated to contain over 1 mgrm. Fe. The experiment with 5 mgrms. Fe was repeated except that 5 cc. NaOH were added to the $NaHCO_3$ filtrate; no precipitate resulted in the cold, but about 0·5 mgrm. Fe as $Fe(OH)_3$ precipitated on boiling. The precipitation was still incomplete for about 0·5 mgrm. Fe was found in the solution on acidifying and adding NH_4OH.

0·5 mgrm. Fe as $FeSO_4$, and in a second experiment as $FeCl_3$, was boiled with 30 cc. 10 per cent $NaHCO_3$, for one minute, the mixtures were poured through filters, cooled, and H_2S was passed in for about three seconds; the solutions became dark coloured at once. After about five minutes the mixtures were filtered; the filtrates were clear, with a faint greenish shade corresponding to an insignificant amount of iron; more H_2S caused no further

* From the *Journal of the American Chemical Society*, xxx., No.4.

darkening. The experiment with 0·5 mgrm. Fe as $FeCl_3$ was repeated, except that the $NaHCO_3$ solution was not cooled before passing in H_2S; a dark green solution was obtained which ran through the filter, showing that it is better to add the H_2S in the cold. To this solution (which was now cold) was added 2 or 3 mgrms. Fe as $FeSO_4$; on filtering after several minutes the filtrate was nearly colourless, and more H_2S gave no precipitate nor colour.

*P. 58*h*, N. 2: *Behaviour of Uranyl Salts in Strong Sodium Hydrogen Carbonate Solution, and on Passing in* H_2S.—50 mgrms. U as UO_2Cl_2 (and in another experiment as $UO_2(NO_3)_2$) were dissolved in a little water and added to a solution containing 5 grms. $NaHCO_3$, the final volume being 50 cc.; the mixture was boiled for five minutes; no precipitate separated. The experiment was repeated with 5 mgrms. U as UO_2Cl_2 in a volume of 30 cc. and 10 cc. 10 per cent NaOH was added to the 10 per $NaHCO_3$ solution after boiling; no precipitate separated.

To a mixture containing 100 mgrms. Be and 20 mgrms. U dissolved in 50 cc. 10 per cent $NaHCO_3$ was added 1 grm. NH_4Cl, and the mixture was boiled; no precipitate separated.

A mixture containing 100 mgrms. Be and 10 mgrms. U (but no iron) dissolved in 30 cc. 10 per cent $NaHCO_3$ solution was saturated completely with H_2S gas; no precipitate separated.

The Separation of Uranium and Beryllium by Potassium Ferrocyanide.—15 and 25 mgrms. Be as chlorides in HCl solution were evaporated almost to dryness, 10 cc. saturated NaCl solution added, the mixture was cooled and 5 cc. 10 per cent $K_4Fe(CN)_6$ solution were added; clear solutions resulted which were blue coloured, owing to the presence of a small amount of iron in the beryllium; after standing several hours a gelatinous light coloured precipitate had separated in the experiment with 25 mgrms. Be. The experiment was repeated with mixtures of 1 mgrm. U as $UO_2(NO_3)_2$, and with 5, 10, 15, and 20 mgrms. Be; a dark red colour appeared at once in the experiments with 5 and 10 mgrms. Be, and on standing dark red precipitate settled out. In the experiments with 15 and 20 mgrms. the colour was somewhat obscured owing to the presence of the iron. The experiment with 20 mgrms. Be and 1 mgrm. U was repeated, except that the iron was first removed from the beryllium solution by P. 52; a good test for uranium was then obtained.

P. 61, N. 1: *Solubility of Manganic Peroxide in* HNO_3 *in the Presence of Filter-paper.*—500 mgrms. Mn as $Mn(NO_3)_2$ were treated by P. 52; the $MnO(OH)_2$ precipitate was boiled with 30 cc. HNO_3 (1·20) in a covered casserole for several minutes; very little of the precipitate dissolved. The experiment was repeated except that the filter, as well as the precipitate, was treated with the nitric acid; on boiling, the filter disintegrated and the precipitate dissolved completely in ten or fifteen minutes. The last experiment was repeated with HNO_3 (1·42); nearly all of the precipitate dissolved after five minutes' boiling, and the remainder on standing over night. The solution obtained in the last experiment was evaporated to a small volume and diluted to about 15 cc.; the paper was filtered off, and the filtrate was treated by P. 61; the manganese was completely precipitated.

P. 62, N. 2: *Precipitation of Manganese by Chloric and Nitric Acids.*—See Hannay (*Journ. Chem. Soc.*, 1878, xxiii., 269); Ford (*Trans. Inst. Min. Eng.*, ix., 397).

P. 62, N. 3: *Separation of Other Elements from Manganese by Chloric and Nitric Acids.*—See T. A., Nos. 117, 118.

Iron.—100 mgrms. Mn as $MnCl_2$ and 1 mgrm. Fe as $FeSO_4$ were treated by P. 52 and P. 61, and the filtrate was tested for iron by P. 64; a good test was obtained. 500 mgrms. Mn as $MnCl_2$ and 1 mgrm. Fe were treated by P. 52 and then by P. 61; the precipitate and filtrate were tested for iron by evaporating with excess of HCl, diluting, and adding KSCN solution; a distinct test for iron was obtained in the filtrate, but much more iron was found in the precipitate than in the filtrate.

Titanium.—A mixture containing 500 mgrms. Mn and 1 mgrm. Ti as nitrates was treated by P. 61; the filtrate was evaporated and made alkaline with NH_4OH; no precipitate separated, proving that the titanium had been completely carried down with the MnO_2. This experiment was repeated with 5 and with 50 mgrms. Ti; the result was the same in each case. The experiment was repeated with 50 mgrms. Mn and 50 mgrms. Ti; the precipitate was analysed by *P. 62*a*, and about half the titanium found in it; the filtrate was evaporated almost to dryness; some of the titanium separated during the evaporation as a white precipitate, which did not dissolve readily in HCl, but did in HF. The experiment was repeated with 50 mgrms. Ti in the absence of manganese; no precipitate separated on adding $KClO_3$, nor on evaporating to 5 or 10 cc.

Zirconium.—A mixture containing 500 mgrms. Mn and 2 mgrms. Zr as nitrates was treated by P. 61; the filtrate was evaporated and made alkaline with NH_4OH; only a very small precipitate separated, showing that not quite all of the zirconium had been carried down. The experiment was repeated with 50 mgrms. Zr; the filtrate was found to contain 15 or 20 mgrms. Zr. The manganese precipitate was treated by *P. 62*a*; the rest of the zirconium was recovered.

Vanadium.—A mixture containing 250 mgrms. Mn as nitrate and 1 mgrm. V as Na_3VO_4 was treated by P. 61; the filtrate was treated by *P. 58*e* to test for vanadium; no trace of vanadium was found. For the fact that vanadium is not carried down by manganese in the Na_2O_2 procedure see C. E., P. 52, N. 11. The experiment was repeated with 250 mgrms. Mn and 10 mgrms. V; a mere trace of vanadium was found. The experiment was repeated with 50 mgrms. Mn and 10 mgrms. V; not more than 1 or 2 mgrms. V were found in the filtrate. The experiment was repeated with 26 mgrms. Mn and 10 mgrms. V; the filtrate contained not more than 2 or 3 mgrms V.

Uranium.—A mixture of 250 mgrms. Mn as $Mn(NO_3)_2$ and 1 mgrm. U as $UO_2(NO_3)_2$ was treated by P. 61; the filtrate was tested for uranium by *P. 58*d*; a good test for uranium was obtained.

Thallium.—15 mgrms. Tl as $Tl(OH)_3$ were treated by P. 61; no precipitate separated. 200 mgrms. Mn as $MnCl_2$ and 1 mgrm. Tl as $TlCl_3$ were treated by this procedure. The filtrate was tested for thallium by the regular procedure; a good test was obtained. The precipitate was dissolved in HCl (1·12) and treated by *P. 65*a*—*d*; a very small precipitate of TlI was obtained, which was estimated to contain not more than 0·1 mgrm. Tl, showing that thallium is not carried down with the manganese.

The Test for Titanium with H_2O_2 *in the Presence of Iron, Cobalt, or Nickel.*—500 mgrms. Fe as $FeCl_3$ were evaporated with a large excess HNO_3 (1·42) to 5 cc.; the dark red solution was diluted to 40 cc.; it became almost colourless. The solution was divided into two parts, and to one of these 0·5 mgrm. Ti as $TiCl_4$ solution was added; to both parts 2 or 3 cc. H_2O_2 were added; in both a deep yellow colour very clearly indicated the presence of titanium. The experiment was repeated, except that the HNO_3 solution was diluted to about 15 cc.; the test for titanium was distinct, although the ferric solution was not quite colourless.

500 mgrms. Co as $Co(NO_3)_2$ were evaporated with excess HNO_3 (1·42) to 5 cc.; the red solution was diluted to 15 cc. and divided into two parts, to one of which 1 mgrm. Ti was added; 2 cc. 3 per cent H_2O_2 were added; the solution containing titanium acquired a reddish colour of a distinct yellow tinge.

The experiment was repeated with 500 mgrms. Ni as $Ni(NO_3)_2$; the green colour of the nickel solution containing titanium changed to olive when the H_2O_2 was added.

P. 62, N. 1: *Confirmatory Test for Manganese with Lead Dioxide.*—To 1 grm. PbO_2 and 10 cc. HNO_3 (1·20) in a casserole were added in separate experiments 0·5, 0·2, 0·1, and 0·02 mgrm. Mn as $MnCl_2$. The mixtures were boiled gently for about two minutes, in covered casseroles, and then poured into test-tubes; after the PbO_2 had settled the colour of $KMnO_4$ could be clearly seen, even in the last experiment. The experiment was repeated without adding $MnCl_2$; a perfectly colourless solution was obtained. The series of experiments was repeated, except that HNO_3 (1·42) was used; the colour of $KMnO_4$ was easily distinguished in each case, but was not so pronounced as with the more dilute acid. Moreover, on standing the pink colour faded slowly in the concentrated HNO_3.

To determine whether the test would be satisfactory when the manganese was initially present as MnO_2, 1 mgrm. Mn as $MnCl_2$ was treated by P. 61, and the precipitate collected on an asbestos filter. About one-tenth of it (*i.e.*, 0·1 mgrm. Mn) was treated by P.-62; the permanganate colour was very distinct.

*P. 62*a*, N. 1: *Separation of Titanium and Zirconium from Manganese by Ammonia.*—To a mixture containing 50 mgrms. Mn and 50 mgrms. Ti as nitrates, and 4 cc. HCl (1·12) in 50 cc. was added NH_4OH (0·96) very carefully until the mixture was barely alkaline to litmus-paper; a white precipitate formed. Two drops more NH_4OH (0·96) were added, and the mixture was heated on a steam-bath for ten minutes; the precipitate remained white. The solution was filtered. The filtrate was tested for titanium by adding more ammonia; no more precipitate separated at once, showing that the precipitation of the titanium was complete. The precipitate was dissolved in hot HCl, the process was repeated, and the filtrate was tested for manganese by adding $(NH_4)S_2$; a precipitate estimated to contain 3 or 4 mgrms. Mn separated. The titanium precipitate was again dissolved and treated in the same way; no manganese was found in the filtrate, showing that the manganese had been completely removed in two separations. The experiment was repeated except that the two drops of ammonia in excess were not added; only about two-thirds of the titanium precipitated. The experiment was repeated, except that NH_4OH was added until the odour after shaking was distinct; the NH_4OH precipitate was brown, showing the presence of a large amount of manganese. The last experiment was repeated, except that 15 grms. NH_4Cl were also added; the NH_4OH precipitate was white, and contained only about 1 mgrm. Mn. These experiments prove that a satisfactory separation is obtained only when the hydroxide-ion concentration is very small.

A solution containing 500 mgrms. Mn and 1 mgrm. Ti as nitrates, and 10 cc. HCl (1·12) in 60 cc., was made barely alkaline to litmus-paper by means of NH_4OH (0·96), an excess of two drops NH_4OH was added, and the mixture was heated for ten minutes on a steam-bath; the precipitate was dark coloured. The precipitate was dissolved in hot HCl and the separation repeated; a small white precipitate resulted, which was practically free from manganese. It was dissolved in HCl, and a little 3 per cent H_2O_2 was added; the solution became deep yellow, showing the presence of titanium. The experiment was repeated with 500 mgrms. Mn and 2 mgrms. Zr as nitrates; the results were the same. The final white precipitate was dissolved in hot HCl, the solution was evaporated to a few drops, and a piece of turmeric-paper was dipped in it and dried over a small flame; it turned pink, proving the presence of zirconium.

Separation of Zirconium from Manganese by Ammonium Acetate.—A solution of 500 mgrms. Mn and 5 mgrms. Zr in 10 cc. HCl (1·12) was just neutralised with NH_4OH; 2 cc. 30 per cent acetic acid were added, the mixture was diluted to 50 cc., 6 cc. 50 per cent ammonium acetate were added, and the mixture was boiled; the precipitate was brown, showing that several mgrms. of manganese were present and that the separation was unsatisfactory.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, November 27th, 1908.

Dr. CHARLES CHREE, F.R.S., President, in the Chair.

THE PRESIDENT announced that the Council had appointed Mr. S. W. J. Smith to succeed the late Prof. Cassie as an Honorary Secretary of the Society.

Mr. R. L. Jones was elected a Fellow of the Society.

A paper entitled "*A Graphic Method of dealing with Refracting Surfaces*" was read by Mr. H. S. ALLEN.

In this paper a graphic method is given for finding the cardinal points for combinations of coaxial refracting surfaces. The method may be applied to such cases as that of two thin lenses a finite distance apart, two refracting surfaces forming a thick lens, or to the general case of the combination of two lens systems. Through the second focal point of the first system, set off a line $F_1'E_1$ at right angles to the axis, equal to the second focal length of that system. Draw F_2E_2 similarly through the first focal point of the second system equal to the first focal length of that system. F'_1E_2 and F_2E_1 intersect in C a point in the plane of the optic centre of the combination. The images of C formed by the two systems determine the principal planes of the combination. By tracing the rays CE_1 and CE_2 through each system a simple construction is obtained for the cardinal points and focal lengths. The method avoids the use of continued fractions, and may be of value for investigating the way in which the properties of a combination are affected by making given alterations in the character or separation of the components. It affords non-mathematical students a means of realising the existence of the cardinal points for a complicated system of refracting surfaces.

Dr. ECCLES remarked that the author's treatment of refracting surfaces was limited to spherical surfaces and to the direct incidence of pencils of infinitesimal aperture. If the treatment were extended to the discussion of oblique pencils and the construction of caustics it would be very valuable. The author's method, as developed to the present, consisted of applying the well-known graphical construction for a harmonic mean, to the successive lenses or spherical surfaces met with in the coaxial system of lenses or surfaces under consideration. Now the harmonic construction just mentioned is a particular case of the general graphical connection between two collinear projective (or homographic) ranges of points; and therefore the speaker preferred, in his own teaching of geometrical optics, to use general projective constructions rather than this harmonic construction. As a fact, the general projective method would be felt to be the easier of the two, by any one familiar with the fundamental principles of projective geometry, or, as it is sometimes called, "geometry of position." From the point of view of projective geometry the object-points and the image-points on the axis of a refracting (or reflecting surface), since they have a true one-one correspondence (as shown by their algebraic homographic connection), constitute two collinear projective ranges of points. Two such ranges can be graphically correlated, it is known, by at most three projections. The rays used in the first and last of these projections may be interpreted optically as rays of light through a source of light and through its image. The intermediate projection gives the central point on which the author lays stress. If this point be taken at infinity the planes of unit magnification become defined. Since any number of successive projections at successive surfaces leave the ultimate range of image-points homographic with the original range of object-points, it is proved that any lens system whatever of the kind specified by the author can be summarised graphically by three projections. Moreover, no graphical construction can be

obtained which is not comprehended in the general projective method just sketched. In treatises on projective geometry the properties of these projective ranges and pencils are demonstrated, and they need merely be translated into optical language to give many beautiful and powerful graphical optical theorems. There is reason to think that caustics can be approached along these lines. The speaker expressed the hope that the author would devote still further effort to these much desired simplifications of geometrical optics.

Dr. A. Russell congratulated the author, and indicated a simple graphical method of determining the focal length of two thin lenses in contact when the focal length of each was known.

Prof. C. H. Lees thought the graphical method described by the author was an improvement upon algebraic methods.

A paper on "*An Accurate Method of Measuring Moments of Inertia*," by the late Prof. W. Cassie, was read by the President.

In this method use is made of the periods of small oscillations of a balance-beam. The method consists in taking as standard moment of inertia a known mass hung from the knife-edge of a balance and comparing others with it. The time of swing is taken with the standard mass in one pan and a counterpoise in the other. The body whose moment of inertia is required is attached to the beam in such a manner that the coefficient of the directive couple is unaltered and the time of swing is determined without weights in the pans. From these times, with a knowledge of the length of the beam and the masses used, the moment of inertia required is easily calculated. In the paper the method of attaching the body of unknown moment of inertia to the balance-beam is described fully.

The Secretary read a letter from Prof. W. Stroud describing a method of determining moments of inertia in use in the University of Leeds. In this method two vibrators perpendicular to one another are used, and they can be arranged to produce a Lissajou figure. The horizontal vibrator carries a vertical slit and *vice versa*; and as the two slits are placed one behind the other, a small spot of light can be seen through the combination. The vertical vibrator consists of a stout metal framework supported by a torsion wire, and carrying a platform upon which the body whose moment of inertia is required can be placed. The time of swing of this vibrator is then adjusted (by the addition of rings of known moment of inertia) to be equal to that of the horizonal vibrator, so that the Lissajou figure is invariable. The body is then placed upon the platform, and inertia rings are removed till the Lissajou figure is reproduced or nearly so. Then, accurate observations are taken of the number of oscillations required to complete a cycle with inertia rings slightly below and slightly above the correct value, which is got by interpolation. The vibrators are kept in continuous oscillation by an electromagnetic make and brake.

Prof. C. H. Lees said that Prof. Stroud's method did not seem capable of giving very accurate results on account of want of homogeneity of the rings.

Dr. A. Russell asked if it was necessary to take account of changes in barometric pressure in accurate work on moments of inertia.

The Chairman remarked that in delicate work attention must be paid to the effects of changes in barometric pressure.

Mr. S. Russ read a paper "*On the Diffusion of Actinium and Thorium Emanations.*"

Experiments were described in which the emanation of actinium was allowed to diffuse into the following gases: —Air, hydrogen, carbon dioxide, sulphur dioxide, and argon. The diffusion coefficients of the emanation in these gases agree in general with those calculated by means of Graham's Law, making use of the diffusion coefficient in air, The variation with pressure of the diffusion coefficient of the actinium emanation in air was shown to be quite in accordance with the ordinary gas laws, down to a few centimetres pressure; the product the pressure and diffusion cofficient remaining practically constant. Experiments under identical experimental conditions with thorium emanation over a similar range of pressure also yield nearly a constant value for the product of pressure and diffusion coefficient. The ratio of the two constants thus obtained leads directly to a ratio of the molecular weights of the two emanations, the result being that thorium emanation appears to have about 1·4 times the molecular weight of actinium emanation.

Prof. C. H. Lees pointed out that the low values for the molecular weights of the emanations had all been obtained by allowing the emanations to diffuse into light gases. Prof. Pickering had allowed them to diffuse into mercury vapour, and obtained a value about 230.

A paper, "*On the Elliptic Polarisation produced by the Direct Transmission of a Plane Polarised Stream through a Plate of Quartz cut in the Direction Oblique to the Optic Axis, with a Method of determining the Error of a Plate supposed to be Perpendicular to the Axis*," by Mr. J. Walker, was taken as read.

When a stream of light polarised in an azimuth α with respect to the principal section traverses a plate of quartz cut obliquely to the optic axis, the polarisation of the emergent stream is given by equations (3), in which $\tan\gamma$ is the ratio of the elliptic vibrations and θ determines the azimuth of the plane of maximum or minimium polarisation, according as γ is numerically less or greater than $\pi/4$; the quantities R, Δ occurring in these equations are connected with the ellipticity of the polarisation of the streams of permanent type within the crystal and the relative retardation, δ, introduced by the passage of equations (1).

Expressing $\tan(\theta - \alpha)$ and $\sin 2\gamma$ in terms of $\tan R$, we find that they both take the form—

$$\frac{2(a\tan R + b\tan^2 R}{1 + c\tan^2 R},$$

the maximum and minimum values of which are $a\tan R_m$, occurring when—

$$\tan R = \tan R_m = \frac{b \pm \sqrt{b^2 + a^2 c}}{ac}.$$

From these equations the changes in the character of the emergent stream may be traced as R increases through a half period, corresponding to the variation of δ through a complete period.

Writing $\tan\psi = \tan 2\alpha/\sin 2\beta$, it may be noted that the emergent light is plane polarised in the primitive plane when $\delta = 2n\pi$, and is again plane polarised in a plane symmetrical to the primitive plane with respect to the principal section when $\delta = (2n+2)\pi - 2\psi$, and that $\sin 2\gamma$ attains its maximum and minimum values when $\delta = (2n+1)\pi - \psi$ and $(2n+2)\pi - \psi$. Also that $\sin 2\gamma$ can only become numerically equal to unity, if—

$$\cos 2\alpha = \pm\tan 2\beta,$$

and hence, except in these cases, θ always gives the plane of maximum polarisation and the light is never circularly polarised.

On the other hand, maximum and minimum values of $\tan 2(\theta - \alpha)$ only occur, if $\cos^2 2\alpha > \tan^2 2\beta$, and when this is the case, writing—

$$\cos^2 2\alpha - \tan^2 2\beta = \cos^2\chi,$$

these values are $\tan(\chi - 2\alpha)$ and $-\tan(\chi - 2\alpha)$.

Thus as R increases the plane of maximum polarisation rotates continuously, if $\cos^2 2\alpha < \tan^2 2\beta$, while if $\cos^2 2\alpha > \tan^2 2\beta$ it oscillates between two extreme positions.

This is a generalisation of a condition given by Monnory for the special case in which $\alpha = 0$. When $\cos^2 2\alpha = \tan^2 2\beta$, θ increases by a right-angle only, as δ changes by a complete period; the light is circularly polarised when $\tan R = \cot 2\alpha$, and while for values for R less than that given by this equation, θ gives the plane of maximum polarisation, for greater values it determines the plane of minimum polarisation.

In § 6 the expression is obtained for the intensity when the light is analysed in any azimuth, and the result is applied to the case in which white light passes and is subsequently examined with a spectroscope—an investigation rendered necessary by the difference in the accounts of the phenomenon given by Cruellebois and Beaulard. If the light emergent from the quartz be investigated with a Savart's analyser, it is clear that the bands will only disappear when the light is plane polarised in a plane parallel to the principal section of either plate of the analyser. Now it is shown in an early part of the paper that the light on leaving the quartz is plane polarised (1) in the primitive plane, when $\delta = 2n\pi$, (2) in a plane making an angle $n\pi - \alpha$ with the principal section, when—

$$\tan \frac{\delta}{2} = -\tan 2\alpha/\sin 2\beta.$$

It thus follows, that if the light be initially polarised in a plane parallel to the principal section of either plate of analyser, *i.e.*, in a plane such that the bands disappear when the quartz plate is away, there will be a disappearance of the bands when $\delta = 2n\pi$, and that if the principal section of the quartz plate bisect the angle between the principal sections of the Savart's compound plate, there will be in addition a further disappearance of the bands whenever $\delta = (2n+1)\pi$.

This leads to a method of determining the error of a plate supposed to be perpendicular to the optic axis, that is described at the end of the paper.

A paper by Mr. W. C. M. Lewis, entitled "*An Experimental Investigation of Gibbs's Theory of Surface-concentration regarded as the Basis of Adsorption*," was taken as read.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 19, November 9, 1908.

Products of the Reaction of Sodamide on Ketones.—A. Haller and Ed. Bauer.—When a benzene or toluene solution of benzophenone is boiled with sodamide, a copious white precipitate is formed, sometimes preceded by an intense blue coloration, and the sodamide slowly dissolves with or without the formation of a deposit. The coloration is undoubtedly due to the action of small quantities of sodium, retained by the amide, on benzophenone, for when no alkali metal is present sodamide gives no coloration with benzophenone; moreover, sodium alone, when heated with benzophenone, gives an intense coloration. The white precipitate is not formed when an absolutely anhydrous solvent is employed; if it is not formed at once the addition of a few drops of water will hasten its appearance. The precipitate seems to consist of a sodium amido compound of benzophenone of formula—

$$C_6H_5C\begin{matrix}ONa\\C_6H_5\\NH_2\end{matrix}.$$

The reactions are—

$$C_6H_5.CO.C_6H_5 + NH_2Na = C_6H_5.C\begin{matrix}ONa\\C_6H_5\\NH_2\end{matrix},$$

$$C_6H_5.C\begin{matrix}ONa\\C_6H_5\\NH_2\end{matrix} + H_2O = C_6H_5CONH_2 + C_6H_6 + NaHO.$$

This seems to be a general reaction for all aromatic ketones; anthraquinone, however, does not give it.

Ionisation by Phosphorus and Phosphorescence.—Léon and Eugène Bloch.—By means of a current of gas the phosphorescence may be separated from phosphorus by a perfectly dark space. The phosphorus sometimes retains a slight phosphorescence, but this may be altogether extinguished by increasing the flow of gas. The authors experiments show that phosphorescence, ionisation, and ozone are all formed in the same region, which may be separated from the element when the current of gas is rapid enough. This shows that these phenomena are not produced by the direct oxidation of solid phosphorus, but by the oxidation of a substance which emanates from the phosphorus and is carried away by the current of gas. This substance is very probably phosphorous anhydride. If a current of gas is passed rapidly enough through a tube containing phosphorus to render it perfectly dark, and is then suddenly cut off, phosphorescent specks are seen. They disappear when they come in contact with one another. This phenomenon may be explained by supposing that the dark tube contains unoxidised phosphorous anhydride which is spontaneously inflammable. Hence phosphorescence, ionisation, and formation of ozone are not due to the oxidation of phosphorus, but of phosphorous anhydride.

True Atomic Weight of Silver from Stas's Experiments.—Louis Dubreuil.—Generally speaking, it is impossible to deduce the true value of an atomic weight by determining a single analytical ratio. The application of the method of the least squares enables the most probable value of all the atomic weights found in each experiment to be calculated. By comparing the values thus obtained the true atomic weight can be deduced, the number being more accurate the greater the number of methods employed. Applying these principles to the case of silver it is found that the true atomic weight does not differ from 108 by more than +0·0079.

Alloys of Silicon and Silver.—G. Arrivaut.—The author has investigated the formation of compounds of silicon with silver by Tammann's method of thermic analysis. He finds that the curve of the liquid phase is composed of two branches, starting from the melting-points of silicon (1415°) and silver (950°) respectively, and cutting at 800°, at a point corresponding to a mixture containing about 5 per cent of silicon. The curve of the solid phase consists of a straight line passing through the eutectic point, and reaching from 0 to 90 per cent of silicon. Thus, silicon and silver form no compound. As to the miscibility in the solid state the duration of eutectic crystallisation points to the existence of mixed crystals of silicon and silver saturated from 10 per cent of silver.

Identity of Ilicic Alcohol with α-Amyrine.—E. Jungfleisch and H. Leroux.—The identity of ilicic alcohol with α-amyrine may be proved by the comparison of the physical properties of the two substances and of their acetic and benzoic ethers. The benzoate is dimorphous, existing as long silky needles and also in the form of prisms. A mixture of the two forms can exist for several days, but the prismatic form seems to increase slowly at the expense of the other.

New Method of Cyclisation of α-Methyl Sparteïn.—Amand Valeur.—α-Methyl sparteïn readily fixes bromine and iodine, the fixation in the case of iodine being accompanied by an isomerisation. In diiodo-α-methyl sparteïn one of the iodine atoms is attached to the nitrogen, and hence the compound is the iodomethylate of an iodated base. The reduction of the diiodo-compound gives the iodomethylate of isosparteïn, and hence the former is the iodomethylate of iodoisosparteïn.

TO CORRESPONDENTS.

G. J. Woods.—Chrome alum dissolves in about 6 or 7 parts of cold water. Consult A. M. Comey's "Dictionary of Chemical Solubilities," published by Macmillan and Co.

MEETINGS FOR THE WEEK.

Monday, 21st.—Faraday Society, 8. "Influence of Cheap Electricity on Electrolytic and Electrothermal Industries," by E. A. Ashcroft.

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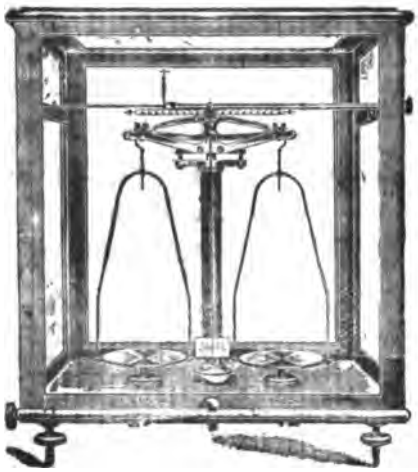

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ON SCANDIUM.*

By Sir WILLIAM CROOKES, D.Sc., F.R.S.

(Concluded from p. 297).

Scandium Ortho-toluate,

$\left(C_6H_4<\begin{matrix}CH_3\\COO\end{matrix}\right)_2ScOH,3H_2O =$

$= (C_8H_7O_2)_2ScOH,3H_2O = C_{16}H_{15}O_5Sc,3H_2O.$

When scandium hydroxide is boiled with an excess of aqueous ortho-toluic acid, a salt is formed with the composition given above. It is a white crystalline powder insoluble in water.

The same salt is formed by mixing solutions of ammonium ortho-toluate and scandium nitrate. It forms a voluminous white curdy precipitate, insoluble in hot or cold water. It contains 3 molecules of water, which are not driven off at 150°.

Analysis.

12·316 grs. of scandium ortho-toluate dried *in vacuo* over sulphuric acid left on ignition 2·171 grs. of scandia, = 1·4059 grs. of scandium.

		Theory.		Experiment.
Sc		44·100	11·45	11·42
C_{16}	192·000			
H_{15}	15·120	341·168	88·55	88·58
O_5	80·000			
$3H_2O$	54·048			
	385·268	100·00		100·00

It is seen that this ortho-toluate has the same percentage composition as the phenyl-acetate, but differs in structural constitution.

Basic Scandium Ortho-toluate.

When scandium hydroxide is boiled with solution of ortho-toluic acid, keeping the hydroxide in slight excess, a basic scandium ortho-toluate is formed. It can be decanted easily from the excess of hydroxide, which is of greater density; it is anhydrous and has the composition—

$$2C_6H_4<\begin{matrix}CH_3\\COOScO\end{matrix} + Sc(OH)_3 = 2C_8H_7O_3Sc + Sc(OH)_3.$$

This basic ortho-toluate is a white powder, insoluble in water and alcohol, but easily soluble in dilute acids.

Analysis.

1. 6·402 grs. of basic scandium ortho-toluate, dried over sulphuric acid *in vacuo*, left on ignition 2·711 grs. of scandia, = 1·7556 grs. of scandium.
2. 7·631 grs. of basic scandium ortho-toluate left on ignition 3·219 grs. of scandia, = 2·0846 grs. of scandium.

		Theory.		Experiment.	
				1.	2.
Sc_3		132·300	27·25	27·42	27·32
C_{16}	192·000				
H_{17}	17·136	353·136	72·75	72·58	72·68
O_9	144·000				
	485·436	100·00		100·00	100·00

* A Paper read before the Royal Society, April 30, 1908.

The above basic salt is formed by the union of two molecules of the ortho-toluate, $C_8H_7O_3Sc$, with one molecule of scandium hydroxide, $Sc(OH)_3$.

Scandium Meta-toluate,

$\left(C_6H_4<\begin{matrix}CH_3\\COO\end{matrix}\right)_2ScOH,3H_2O =$

$= (C_8H_7O_2)_2ScOH,3H_2O = C_{16}H_{15}O_5Sc,3H_2O.$

This salt is formed by double decomposition between ammonium meta-toluate and scandium nitrate solutions. It comes down as a voluminous white curdy precipitate, insoluble in water.

Analysis.

15·702 grs. of the meta-toluate, dried *in vacuo* over sulphuric acid at the ordinary temperature, left on ignition 2·673 grs. of scandia, = 1·7310 grs. of scandium.

		Theory.		Experiment.
Sc		44·100	11·45	11·02
C_{16}	192·000			
H_{15}	15·120	341·168	88·55	88·98
O_5	80·000			
$3H_2O$	54·048			
	385·268	100·00		100·00

Scandium Para-toluate,

$\left(C_6H_4<\begin{matrix}CH_3\\COO\end{matrix}\right)_2ScOH,3H_2O =$

$= (C_8H_7O_2)_2ScOH,3H_2O = C_{16}H_{15}O_5Sc,3H_2O.$

Scandium para-toluate is formed by double decomposition in the same manner as the ortho-toluate, and has the same composition. It separates with three molecules of water, which are not driven off at 150°. It is a white curdy precipitate insoluble in hot or cold water.

Analysis.

12·332 grs. of scandium para-toluate on ignition left 2·141 grs. of scandia, = 1·3865 grs. of scandium.

		Theory.		Experiment
Sc		44·100	11·45	11·24
C_{16}	192·000			
H_{15}	15·120	341·168	88·55	88·76
O_5	80·000			
$3H_2O$	54·048			
	385·268	100·00		100·00

Scandium Phenyl-acetate,

$(C_6H_5CH_2COO)_2ScOH,3H_2O = C_{16}H_{15}O_5Sc,3H_2O.$

Scandium hydroxide is readily attacked by aqueous phenyl-acetic acid, with the formation of a white insoluble phenyl-acetate. The salt thus prepared has, however, a tendency to become more basic, and a better way to prepare it is by double decomposition between ammonium phenyl-acetate and scandium nitrate. When moderately dilute and hot solutions of these salts are mixed and the whole boiled, the phenyl-acetate comes down chiefly on the sides and bottom of the beaker, and adheres to the glass with great pertinacity. It can only be removed with difficulty and risk of breakage. The deposit on the glass looks like warty concretions and under a high power appears crystalline. It is almost insoluble in hot or cold water, and is insoluble in alcohol.

When double decomposition between scandium nitrate and ammonium phenyl-acetate is effected in the cold, the salt comes down as a white curdy precipitate which does not adhere to the glass, and can be washed easily with warm water; boiling water, however, causes it to cohere with contraction. The dry salt when heated softens and can be pressed into a pasty mass. It does not fuse at a temperature at which decomposition begins.

Analysis.

1. 13·584 grs. of scandium phenyl-acetate precipitated from hot solutions and washed with boiling water, filtered, and dried at 100° till the weight was constant, yielded on ignition 2·396 grs. of scandia, = 1·5516 grs. of scandium.
2. 9·458 grs. of scandium phenyl-acetate precipitated from cold solutions, washed, and dried at 100° to a constant weight, gave on ignition 1·671 grs. of scandia, = 1·0821 grs. of scandium.

	Theory.		Experiment.	
			1.	2.
Sc	44·100	11·45	11·42	11·44
C_{16} .. .. 192·000				
H_{15} .. .. 15·120	341·168	88·55	88·58	88·56
O_5 .. .. 80·000				
$3H_2O$ .. 54·048				
	385·268	100·00	100·00	100·00

Attempts to estimate the water were unsuccessful, as the phenyl-acetate turned brown and commenced to decompose when heated above 160°, at which temperature the whole of the three molecules of water were not expelled.

Scandium Pyromellitate,

$$C_6H_2\{COOSc(OH)_2\}_4 2H_2O = Sc_4C_{10}H_{10}O_{16},2H_2O.$$

This salt comes down when ammonium pyromellitate is added to a solution of a soluble scandium salt. It is formed also by allowing pyromellitic acid to act on scandium hydroxide with the aid of gentle heat. Scandium pyromellitate is a white amorphous powder, insoluble in water and alcohol, but soluble in dilute acids.

Of the two molecules of water of hydration one is driven off at 100° and the remaining molecule at 140°. The dried salt can be heated to 220° without decomposition.

Analysis.

1. 14·304 grs. of scandium pyromellitate dried at 140° for some hours lost 0·847 gr. of water; when ignited to full redness, treated with nitric acid, and ignited again, it left 6·515 grs. of scandia, = 4·2190 grs. of scandium.
2. 11·485 grs. of scandium pyromellitate dried at 100° lost 0·350 gr. of water, and, when ignited as above, left 5·170 grs. of scandia, = 3·3480 grs. of scandium.
3. 7·614 grs. of scandium pyromellitate heated to 220° for three hours lost 0·464 gr. of water, and, when ignited as already described, left 3·501 grs. of scandia, = 2·2672 grs. of scandium.

	Theory.			Experiment.		
				1.	2.	3.
Sc_4.. ..	176·400	29·47		29·50	29·15	29·78
C_{10} H_{10} O_{16} ..	386·080	64·51		64·58	64·83	64·13
H_2O ..	18·016	3·01	6·02	5·92	3·05	6·09
H_2O ..	18·016	3·01			2·97	
	598·512	100·00		100·00	100·00	100·00

Scandium Camphorate,

$$C_8H_{14}{<}\begin{matrix}COO\\COO\end{matrix}{>}ScOH.$$

The camphorate is prepared by heating scandium hydroxide with a slight excess of camphoric acid and water. The hydroxide is easily attacked and converted into an insoluble camphorate. It may be prepared also by precipitating a solution of scandium nitrate with a solution of ammonium camphorate. In either case, it forms an insoluble white precipitate, which, after thorough washing and drying at 100°, has the composition given above.

Scandium camphorate is anhydrous and insoluble in water and alcohol. It is easily soluble in dilute mineral acids. When dry it is very electrical, and an attempt to powder it sends it flying out of the mortar. If it is put warm between two watch-glasses, and the lower glass is gently rubbed on the outside with the finger, the path of the finger is marked by a flight of camphorate from the lower to the upper glass, where it sticks. This peculiarity makes it not easy to perform the preliminary operations of drying, transferring, and weighing, preparatory to its quantitative analysis.

Gradually heated to redness, the camphorate at first blackens with carbon. At a red heat this burns off, leaving a white residue of scandia.

Analysis.

1. 18·191 grs. of the camphorate, dried at 100°, yielded on ignition 4·772 grs. of scandia, = 3·0902 grs. of scandium.
2. 17·921 grs. of the camphorate yielded on ignition 4·719 grs. of scandia, = 3·0559 grs. of scandium.

	Theory.		Experiment.	
			1.	2.
Sc ..	44·10	17·01	16·99	17·05
C_{10} .. 120·00				
H_{15} .. 15·12	82·99		83·01	82·95
O_5 .. 80·00				
	259·22	100·00	100·00	100·00

Added April 30th, 1908.

Scandium is remarkable inasmuch as its existence was foretold by Mendeleeff, and called by him ekaboron, eight years before its actual discovery by Nilson. (See D. I. Mendeleeff, "A Natural System of the Elements, and its Application to the Indication of the Properties of Undiscovered Elements," *Russ. Chem. Soc. Journ.*, 1871, iii., pp. 25—56). To-day I may be permitted to indulge still further in speculations on the genesis and degradation of the chemical elements—speculations which a few years ago might have been derided even by a learned Society. But the undoubted fact of the production of helium from radium, the probability that chemical elements of high atomic weight are slowly breaking up into bodies of lower atomic weight, and the suspicion that some well-known chemical elements of low atomic weight are degradation products, embolden me to speculate on the past history and genesis of scandium.

In 1898 I brought before the Royal Society a scheme of the arrangement in Space of the chemical elements in which scandium was seen to fall in place between boron and yttrium. I hesitated to introduce ytterbium into the scheme, as its atomic weight was not known with sufficient accuracy to enable it to be properly placed. To-day it is seen to fill a gap below yttrium, and the group is:—Boron, scandium, yttrium, ytterbium. These four elements have a close relationship, thus:—

B	atomic weight	11·0 × 16 = 176·0
Sc	,, ...	44·1 × 4 = 176·4
Y	,, ...	89·0 × 2 = 178·0
Yb	,, ...	173·0 × 1 = 173·0

In reference to this table it must be remarked that the atomic weight of scandium certainly is not known with absolute accuracy, the 0·1 being little more than a guess. Yttrium, moreover, is now seen to be very liable to contain scandium; when Cleve determined its atomic weight it was impossible, without further refined spectroscopic examination—not available at that date—to detect in it the trace of scandium sufficient to lower the atomic weight by one unit. Ytterbium certainly is too low, for Urbain recently has announced the discovery in it of an element of a lower atomic weight; this being removed might raise the atomic

weight of ytterbium a few units. With these reasonable corrections the last column will not be far from the same whole number, 176.

The frequent occurrence of the triad ytterbium, yttrium, and scandium in a very limited group of rare minerals may be explained by assuming the instability of ytterbium, the atom of highest weight, and letting it split into two atoms, when yttrium would be produced. Assume the same action to take place with yttrium, its splitting up again into two atoms would produce scandium; and scandium ("*eka-boron*, next in order to boron") would ultimately form boron by a quadruple sub-division. The comparatively small quantities of ytterbium and scandium in these minerals may easily be accounted for by the assumption that they are more unstable forms of matter than yttrium and boron.

The fact that these bodies give totally distinct line spectra has not the significance it formerly was supposed to have, for now we know of different line spectra given by the same body. Argon, with its red and blue spectra, is a case in point, and Goldstein, as a result of his work on the spectra of Cs, Rb, and K, has come to the conclusion that the property of emitting two separate line spectra is a general one. Moreover, between the line spectra of radium, uranium, and helium—bodies which are supposed to be associated genetically—there is no similarity or line in common.

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W. Crookes. "On Radiant Matter Spectroscopy: Examination of the Residual Glow." *Proc. Roy. Soc.*, February, 1887, vol. xlii., p. 111. "Scandium, either in the form of earth or sulphate, phosphoresces of a very faint blue colour. Addition of lime does not bring out any lines."

"The Arc Spectrum of Scandium, and its Relation to Celestial Spectra." By Sir Norman Lockyer and F. E. Baxandall. *Proc. Roy. Soc.*, February, 1905, vol. lxxiv., p. 538.

G. Urbain (*Journal de Chimie-Physique*, February, 1906, vol. iv., p. 64) gives a passing reference to scandium as follows:—"In none of my fractions have I been able to observe the presence of scandium. I am led to think that this element does not belong to the group of rare earths, properly so-called. Sir W. Crookes (Chemical News, vol. xci., p. 61) thinks he has detected this element in my gadolinia. [Quite true.—W. C.]. The statement is denied by Eberhard (*Zeit. Anorg. Chemie*, xlv., p. 374)."

THE FORMATION OF GRAPHITE BY THE INTERACTION OF MAGNESIUM POWDER AND CARBONATES.*

By H. Russell Ellis, B.Sc., F.C.S.

Winkler, in an investigation of the action of magnesium on the carbonates of the alkaline earths, showed that the carbonates were readily reduced on heating, and amorphous carbon was produced in the reaction (*Ber.*, 1890, xxiii., 2642; see also Moissan, "Electric Furnace," p. 38). The properties of this carbon were not investigated by Winkler or by Maquenne, who also studied the reaction between magnesium and the alkaline earth carbonates.

During an investigation of the action of magnesium powder on various carbonates quantities of carbon were produced, and the object of the present paper is to show that this carbon is a mixture of amorphous carbon and graphite; in some cases it consists almost entirely of graphite. The graphite is non-intumescent.

A mixture of a carbonate and magnesium powder does not require heating before action commences. If such a mixture be ignited in one portion by a match or by means of burning magnesium, a reaction similar to Goldschmidt's occurs, and proceeds throughout the whole mass more or less vigorously; in many cases CO is evolved, which causes much of the contents of the crucible to be ejected; if the metal is of comparatively low boiling-point, this distils off and burns, and so renders the reaction more violent. Since this reaction was accompanied by very high temperature, and since at high temperatures graphite is the stable form of carbon, it was thought that the carbon would be present in this form. In all cases this was found to be so, although amorphous carbon was also present, the graphite being formed as such or formed from amorphous carbon at the extremely high temperature of the reaction.

Detection of Graphite in the Reaction Products.

The contents of the crucible, after a reduction had taken place, were boiled for some time with hydrochloric acid, washed, filtered, and dried, and then treated with fuming nitric acid prepared from boiled sulphuric acid, dry potassium nitrate, and dry potassium chlorate. The mixture was kept cold at first, and then placed on a water-bath, and the temperature slowly brought to 100° C., and the solution evaporated to dryness. If the mixture be made at a high temperature, explosion occurs, and at times the whole mass inflames; by mixing in the cold and warming as described above, in no case was any explosion produced. Small quantities only were used; in nearly every case a deep green coloration was produced at the first treatment, and a yellow powder was left after dissolving the humic acids and potassium chloride in the dry residue; if any black powder was left, this was dried and again treated with the oxidising mixture. The complete conversion of the carbon into humic and graphitic acid seldom required more than one treatment. The graphitic acid obtained readily exploded on heating, forming pyrographitic acid.

Estimation of the Amounts of Graphite and Amorpous Carbon in the Mixture.

In Berthelot's memoir on carbon (*Ann. Chim. Phys.*, 1870, Ser. 4, xix., 392), it was shown that fuming nitric acid attacked amorphous carbon, producing humic acids which were soluble in water, and did not attack graphite. This observation has served as the basis of a method of estimating graphite and amorphous carbon in a mixture of the two.

The total ash is estimated in the mixture by ignition in the muffle furnace; the amorphous carbon quickly burns away, whilst the graphite remains for some time, and only slowly burns. Another portion (0·1 to 0·4 grm.) of the mixture is heated on a sand-bath in a conical flask with

* A Paper read before the Faraday Society, December 15, 1908.

fuming nitric acid for several days until no more brown fumes are evolved, fresh additions of nitric acid being added from time to time. After the oxidation is complete the solution is poured out into an evaporating basin, and evaporated to dryness on the water-bath to remove all the nitric acid. The residue, consisting of humic acid, graphite, and insoluble mineral matter, is extracted with water, and filtered through a tared filter-paper, washed, dried, and weighed. It is then finally burnt in the muffle, the loss of which gives the amount of graphite present. It is essential that the nitric acid be removed, as the mixture is difficult to filter and the filter-paper is attacked.

The method was tested by using a mixture of pure graphite (*Trans. Far. Soc.*, Part I., iv., 70) and amorphous carbon, and it was found to give fairly good results, the graphite being 1 to 2 per cent too high, probably owing to the great difficulty of removing traces of moisture.

This method is very tedious and requires much time, so it was not considered worth while to estimate any of the mixtures obtained. Graphite was obtained from the carbonates of the alkaline earths—ammonium carbonate, cadmium carbonate, and magnesium carbonate—and by the action of magnesium on carbon dioxide.

Reduction of Carbonates of Barium, Calcium, and Strontium.

Mixtures were made in the following proportions:—

(*a*) RCO_3+Mg, (*d*) RCO_3+4Mg,
(*b*) RCO_3+2Mg, (*e*) $RCO_3+3Mg+2C$,
(*c*) RCO_3+3Mg, (*f*) $RCO_3+3Mg+6C$,

and the reaction carried out in open crucibles and in closed iron tins at high temperatures.

In all cases the reaction was violent, especially when carbon was initially present, and in all cases graphite was found in the products of the reaction; in the case of reactions (*c*) and (*d*) very little amorphous carbon was produced.

The reaction between barium and strontium carbonates and magnesium powder when carried out in open crucibles always produced the carbide, nitride, and large amounts of cyanide; when the reaction took place in closed tins, the carbides only were produced. With calcium carbonate no carbide, nitride, cyanamide, or cyanide was produced when open crucibles were used; when the reaction took place at high temperatures in closed tins, calcium carbide only was produced. Since results differing from those of Winkler and Maquenne were obtained, a subsequent paper on this subject will be given.

It is well known that most carbides dissolve carbon and deposit them in the form of graphite; that the graphite was not obtained in this way is evident from the fact that the mass had not a fused appearance, but was a very soft powder.

Action of Magnesium on Ammonium Carbonate, Magnesium Carbonate, and Cadmium Carbonate.

The action was vigorous, and produced graphite, but no nitride, cyanide, or carbide.

Action of Magnesium on Carbon Dioxide.

A rapid stream of dry carbon dioxide was passed over 25 grms. of dry magnesium powder, and this was heated strongly at one point.

After a time the magnesium caught fire and burnt rapidly, producing a black and white residue. This powder was treated with hydrochloric acid, and the carbon filtered and dried; about 2·5 grms. were obtained. Only a very slight amount of carbide was formed, no gas being evolved when the powder above was dropped in water; the smell of acetylene, which is very easily observed from minute quantities of the gas, was, however, noticed.

The black powder contained graphite, but only in small quantity.

Percentage of ash	.. ..	= 6·64
,,	graphite	= 12·09
,,	amorphous carbon . ..	= 81·27

The reaction in this case is comparatively slow, and the mixture was spread out over a long tube, and consequently no very high temperature was attained; on the other hand, when the reduction takes place in crucibles, the heat is concentrated, and a much higher temperature results, and more of the carbon is changed into graphite.

It seems probable that amorphous carbon is first produced by the action of the magnesium on the carbonate, and that more or less of this is converted into graphite owing to the enormous heat evolved.

COLLOIDAL BARIUM SULPHATE.*

(Preliminary Communication).

By ERNEST FEILMANN, Ph.D., F.I.C.

Descriptions of the preparation of colloidal barium sulphate are comparatively rare; G. Buchner (*Chem. Zeitung*, 1893, xvii., 878) describes the precipitation of a gelatinous form of this substance on mixing concentrated aqueous solutions of barium acetate and aluminium sulphate, but on dilution or on standing this reverts to the ordinary form. Neuberg and Neumann (*Biochem. Zeit.*, 1906, i., 166) obtain it by the addition of aqueous sulphuric acid to a methyl alcoholic solution of barium hydroxide, as a jelly which forms a porcelain-like mass on ignition. v. Weinarm (*Zeit. Chem. Ind. Kolloide*, 1908, ii., 89) describes the preparation of numerous salts of the alkali earth and alkali metals, including barium sulphate, in the form of jellies. Some years ago the author of the present communication experimented on the protective action of alkali-casein solutions in the production of colloidal solutions of organic and inorganic substances. For various reasons these were not published at the time; the most various substances, including many of the heavy metals and their more insoluble salts, were thus obtained in solutions of great strength and somewhat surprising stability, and it was thought that a description of such a solution and its method of preparation might not be without interest, especially in view of the ready accessibility in large quantities and cheapness of the materials employed.

Twenty-three grms. of commercial casein were dissolved at below 40° C. in water containing 0·09 grm. of sodium hydroxide and diluted to 230 cc. To this solution was added a solution of 14·2 grms. (0·1 mol.) of anhydrous sodium sulphate in water to 200 cc. To this mixed solution a solution of 24·5 grms. (0·1 mol.) of crystallised barium chloride in water to 100 cc. was then slowly added at 20° C. with vigorous stirring. A thick creamy and almost homogeneous liquid was so obtained, though there was a certain amount of sedimentation on standing. It was precipitated by adding 12 cc. of glacial acetic acid, which caused the formation of a white curd-like precipitate, allowed to stand, and the precipitate washed by repeated decantation till free from acid reaction. It was then stirred with 400 cc. of water and 2 cc. of 15 per cent sodium hydroxide solution at 40° C. for three hours, filtered through an open paper filter, and evaporated to dryness on the water-bath. Before evaporation the liquid had much the appearance of ordinary milk by reflected light; by transmitted light it was transparent in thin layers, showing the peculiar reddish yellow tinge so characteristic of many colloidal solutions. The solid obtained by evaporation was translucent, and of a horny appearance and texture. It was quite soluble in water to a similar solution to the one just described, but on standing it appears to undergo some slight change which renders it necessary to employ a very little dilute alkali in order to obtain a perfect colloidal

* A Paper read before the Faraday Society, December 15, 1908.

solution. On ignition 56·7 per cent of barium sulphate were left. After keeping for six months 2 grms. of the substance dissolved in 70 cc. of water and 0·5 cc. of normal sodium hydroxide solution, leaving only a negligible amount of residue. Under a magnification of 200 diameters particles in rapid movement can just be made out under the microscope, and the appearance of the liquid under magnification is much the same as that of the colloidal solutions of tungsten prepared by Kuzel's method. The solutions are very stable; only a slight amount of sedimentation takes place after standing for many hours, and this is very possibly due to the action of the air, which appears in some way to affect the stability of the solution.

The protective action of organic and other colloids in cases such as the one just considered is one that has not yet been adequately explained, though the phenomenon has been known for a very long time. Some light is thrown on this question by the experiments of Michaelis and Pincussohn (*Biologische Zeit.*, ii., 251), who experimented with colloidal suspensions of indophenol and mastix. These were more stable than suspensions of indophenol alone, and microscopic and optical examination of the mixture led to the conclusion that each particle of indophenol was surrounded by a layer of mastix. There is reason to suppose that the colloidal barium sulphate and other preparations of the present author are in a very similar condition, and that each particle of inorganic material is surrounded by a layer of casein; they behave towards acids and alkalis in a very similar manner to casein itself, being precipitated by the former from solution and re-dissolved by the latter, and this process can apparently be carried on alternatively almost *ab libitum*. These facts suggest that the surface in actual contact with the liquid media consists of casein only, and that it is this surface which conditions the stablity of the solutions obtained. This view is also n accordance with the fact that a certain minimum quantity of casein is in each case necessary to hold the inorganic substance in solution.

A SYSTEM OF QUALITATIVE ANALYSIS FOR THE COMMON ELEMENTS.*

PART III.—ANALYSIS OF THE ALUMINIUM AND IRON GROUPS, INCLUDING BERYLLIUM, URANIUM, VANADIUM, TITANIUM, ZIRCONIUM, AND THALLIUM.

By ARTHUR A. NOYES, WILLIAM C. BRAY, and ELLWOOD B. SPEAR.

(Concluded from p. 303).

Confirmatory Experiments and References (continued).

Complete Precipitation of Thallium by Ammonium Hydroxide.—20 mgrms. Tl as Tl_2SO_4 were treated with HNO_3 and $HClO_3$ by P. 61, and then with NH_4OH by P. 64; a dark red flocculent precipitate separated. This was filtered off, the filtrate was evaporated nearly to dryness, and tested for thallium with KI by *P. 65*d*; no precipitate of TlI separated, showing that thallium had been completely precipitated by the NH_4OH. Since thallous hydroxide is soluble, it follows also that thallous compounds are completely oxidised by HNO_3 and $HClO_3$ in P. 61.

P. 64, N. 2: *Action of Nitric Acid on Potassium Sulphocyanate.*—In each of a number of test-tubes 1 cc. HNO_3 (1·42) was placed; varying amounts of water (from 0 to 15 cc.) and finally 5 cc. 10 per cent KSCN solution were added; in each case a red colour appeared slowly in the cold, more quickly on warming slightly, and more quickly in the more concentrated HNO_3 solutions. The red solutions were boiled in casseroles; the colour disappeared quickly, and the solutions remained colourless on cooling. More KSCN was added; the colour reappeared uickly. The experiments were repeated with 0·1 mgrm. Fe as $FeCl_3$; a red colour appeared at once in the cold, but on boiling this colour also disappeared in a minute or two. To one of these colourless solutions, after cooling, was added KSCN; the red colour appeared at once. To another was added 0·1 mgrm. Fe as $FeCl_3$; the solution remained nearly colourless. These experiments show that on boiling with HNO_3, the KSCN is completely decomposed, and therefore that the red colour due to nitrous acid cannot be eliminated by boiling.

P. 65, N. 2, 5, *and* 6: *Precipitation of Titanium, Zirconium, and Thallium in the Ammoninm Acetate Procedure.*—0·5 mgrm. Ti as $TiCl_4$ in 100 cc. was treated by P. 65; a small precipitate separated, which was proved to contain titanium by *P. 65*b*. The experiment was repeated with 10 and with 500 mgrms. Ti; the filtrates were tested for titanium by evaporating with a little H_2SO_4 to fuming, cooling, and adding H_2O_2; no colour appeared.

A mixture containing 500 mgrms. Fe as $FeCl_3$ and 20 mgrms. Zr as $ZrCl_4$ was treated by P. 65; a large amount of zirconium was found in the precipitate, but on adding NH_4OH to the filtrate a white precipitate was obtained which was estimated to contain 3 to 5 mgrms. Zr. 10 mgrms. Zr as nitrate in HNO_3 solution were treated by P. 65, except that no $FeCl_3$ was added; NH_4OH was added only until a very small precipitate of zirconium hydroxide separated, and the solution was still acid when the ammonium acetate was added; the filtrate from the basic acetate precipitate was made alkaline with NH_4OH to test for zirconium; a white precipitate separated, which was estimated to contain 1 or 2 mgrms. Zr. The experiment was repeated with 1 and 2 mgrms. Zr, except that NH_4OH was added until the solution was neutral; the filtrate was made acid, evaporated to 20 cc., and then made alkaline with NH_4OH; no precipitate separated. The last experiment was repeated with 5 mgrms. Zr as nitrate and 20 mgrms. PO_4 as Na_2HPO_4; no zirconium was found in the filtrate. For the fact that zirconium is not precipitated in the presence of considerable acetic acid, and that it then prevents the complete precipitation of titanium, see Hillebrand (*Bull. U.S. Geol. Survey*, 1900, clxxvi., 72).

20 mgrms. Tl as $TlCl_3$ were treated by P. 65, except that no $FeCl_3$ was added; a brown precipitate separated on boiling and also on adding NH_4OH; the former was two or three times as large as the latter, thus showing that at least 5 mgrms. Tl were not precipitated till the NH_4OH was added. The filtrate was evaporated nearly to dryness; H_2SO_4 was added, and the solution when cold was tested for thallium with KI and Na_2SO_3 by *P. 65*d*; a small precipitate of TlI was obtained which was estimated to contain 0·5 to 1 mgrm. Tl. The experiment was repeated with 1 mgrm. Tl; no precipitate resulted on boiling, nor on adding NH_4OH. The filtrate was proved to contain the thallium. The experiment with 1 mgrm. Tl was repeated, $FeCl_3$ being added as directed in the procedure; no precipitate separated on adding NH_4OH to the filtrate. Both precipitate and filtrate were tested for thallium; about half of the thallium was found in the precipitate and the remainder in the filtrate. The last experiment was repeated with 0·5 mgrm. Tl; a small but distinct test was obtained for thallium both in the precipitate containing the iron and in the filtrate.

*P. 65*a*, N. 1 *to* 3: *Extraction of* $FeCl_3$ *by Ether.*—See Rothe (*Stahl und Eisen*, 1892, xii., 1052; 1893, xiii., 333); Langmuir (*Journ. Am. Chem. Soc.*, 1900, xxii., 102); Kern (*Journ. Am. Chem. Soc.*, 1901, xxiii., 689).

To determine the best concentration of HCl to use in this extraction, a series of experiments was performed as follows:—500 mgrms. Fe as $FeCl_3$ were dissolved in 30 cc. HCl of known specific gravity, and this solution was treated several times with about 35 to 40 cc. ether in a separating funnel as described in the procedure. The amount of $FeCl_3$ extracted in each treatment was estimated by evaporating the ether extract to dryness, adding HCl, and precipitating with NH_4OH. The results are given in the following table. Since the ether used contained initially no HCl, the amount of HCl in the water layer is some-

* From the *Journal of the American Chemical Society*, xxx., No. 4.

what decreased in each treatment. The proportion of $FeCl_3$ extracted in each treatment was 4 to 5 per cent with 8 per cent HCl (sp. gr., 1·04); 95 to 96 per cent with 18 per cent HCl (sp. gr., 1·09); 99 per cent with 22 per cent HCl (sp. gr., 1·11); and 94 per cent with 25 per cent HCl (sp. gr., 1·125).

With the 22 per cent acid (sp. gr., 1·11) the first extraction took out nearly all the $FeCl_3$ (495 mgrms. Fe), the second extraction nearly all of 5 mgrms. Fe remaining, and the third only a fraction of a mgrm. A similar result was obtained when 500 mgrms. Fe as $FeCl_3$ were precipitated with NH_4OH, the precipitate dried between filter-papers, and dissolved in HCl (1·12). This result is in agreement with that of Rothe, who recommends an acid of sp. gr. 1·100 to 1·105, for he shook his ether with HCl of this strength before making the separation.

Molecular Formula of Ferric Chloride in Ether Solution.—See Beckmann (*Zeit. Physik. Chem.*, 1903, xlvi., 860).

Extraction of Thallic Chloride by Ether.—15 mgrms. Tl as $Tl(OH)_3$ were treated by *P. 65*a*; the solution of $TlCl_3$ in HCl (1·12) had a yellow colour, but this layer was colourless after it was shaken with ether; the ether layer had a yellowish colour. The first ether extract contained nearly all the thallium, the second ether extract contained less than 1 mgrm., and the water layer after the two extractions was practically free from thallium.

*P. 65*a*. N. 4: *Behaviour of Titanium, Zirconium, and Uranium in the Ether Treatment.*—A solution of 50 mgrms. Ti as $TiCl_4$ in one experiment, and of 50 mgrms. Zr in another, in 30 cc. HCl (1·11) was shaken with an equal volume of ether; the ether layer was evaporated to dryness in a casserole on a water-bath, HCl (1·20) was added and heated, the solution was made alkaline with NH_4OH; no precipitate separated in either case, showing that no titanium or zirconium had dissolved in the ether layer. In the case of titanium the water layer became reddish yellow owing to the presence of H_2O_2 in the ether, and in the case of zirconium a white precipitate ($ZrCl_4$?) separated in the water layer, but when the ether was expelled by evaporation a clear solution resulted. A white precipitate was also found to result with titanium in an experiment in which 300 mgrms. PO_4 were also present.

For proof that uranium is not extracted, see Kern (*Journ. Am. Chem. Soc.*, 1901, xxiii., 689).

*P. 65*a*. N. 5: *Extraction of Iron by Ether in the Presence of Phosphate.*—500 mgrms. Fe as $FeCl_3$ and 300 mgrms. PO_4 as ammonium phosphate were dissolved in 30 cc. HCl (1·09) and treated with ether (35 cc.) as described in the procedure; about 95 per cent of the iron was extracted in each treatment and the phosphate remained in the water layer.

*P. 65*b*, N. 1: *Nature of the Compound of Hydrogen Peroxide with Titanium.*—In regard to the coloured solution and the preparation of solid TiO_3, see Schönn (*Zeit. Analyt. Chem.*, 1870, ix., 41); Classen (*Ber.*, 1888, xxi., 370; Levy (*Ann. Chim. Phys.*, 1892, [6], xxv., 463); Melikoff and Pissarjewsky (*Ber.*, 1898, xxxi., 953).

Mr. Chas. Field, 3rd, working in this laboratory, performed the following migration experiment. A coloured solution was prepared by rotating for three hours pure TiO_3, which had been dried over P_2O_5, with a normal HNO_3 solution which contained 1 mol. H_2O_2 per litre. The resulting solution was 0·087 molal with respect to TiO_3. A large U tube which was partly filled with normal HNO_3 solution was placed in a thermostat at 25°, and the titanium solution was carefully introduced through a tube at the bottom of the U. The surfaces of contact of the two solutions were sharply marked. After a current had passed for eight hours, one of the surfaces of the red solution had moved 4 cm. upward toward the cathode, and the other boundary had moved away from the anode a nearly equal distance. This proves that the colour is due to a cation containing titanium.

Delicacy of the Hydrogen Peroxide Reaction for Titanium.—0·1 mgrm. Ti as chloride was treated by *P. 65*b*; a distinct yellow colour appeared on adding the H_2O_2 solution.

*P. 65*b*, N2: *Precipitation and Separation of Titanium and Zirconium as Phosphate.*—See Hillebrand (*Bull. U.S. Geol. Survey*, 1900, clxxvi., 75).

0·5 and 1 mgrm. Zr and a mixture of 1 mgrm. Zr and 100 mgrms. Ti as chloride were treated by *P. 65*b*; flocculent precipitates formed in every case within ten minutes, but were more distinct after half an hour. 100 mgrms. Ti alone were treated by *P. 65*b*; no precipitate separated in several hours.

10 mgrms. Zr as chloride were treated by *P. 65*b*; the precipitate was filtered off after half an hour, and the filtrate made alkaline with NH_4OH; a small precipitate estimated to contain about 0·3 mgrm. Zr separated. The result was the same when the filtration was made after twenty hours. The experiment was repeated, except that only 3 or 4 cc. phosphate solution were used; the filtrate contained 0·5 to 1·0 mgrm. Zr after half an hour and after one hour. The experiment was repeated, using about 15 cc. phosphate solution; only 0·1 to 0·2 mgrm. Zr remained in the filtrate after two hours.

To a number of solutions, containing 1 mgrm. Zr as chloride and varying amounts of H_2SO_4 (1·20) in 15 cc., were added 5 cc. 7 per cent $Na_2HPO_4.12H_2O$ solution; with 1 and 2 cc. of acid the solutions became turbid at once, and flocculent precipitates settled out within twenty minutes; with 5 cc. acid the solution remained clear for about ten minutes, but after one hour there was a distinct precipitate; with 10 cc. acid the solution remained clear for a longer time and only a minute precipitate separated in 1·5 hours.

Precipitation of Titanium with Zirconium Phosphate.—10 mgrms. Zr and 10 mgrms. Ti as chlorides were treated by *P. 65*b*; the precipitate when collected on a filter was distinctly yellow, and this colour remained after washing with water for an hour. About 10 cc. 15 per cent HF solution were poured through the filter in a celluloid funnel and the solution was treated again by *P. 65*b*; the phosphate precipitate was white, and the titanium in the solution was estimated from the colour to be about 1 mgrm. 0·1 mgrm. Ti and 100 mgrms. Zr as chlorides were treated by *P. 65*b*; the solution became distinctly yellow on adding H_2O_2, and the filtrate from the phosphate precipitate was also yellow, showing that titanium is not completely carried down by the zirconium. (100 mgrms. Zr alone gave no colour with H_2O_2).

*P. 65*b*, N. 3: *Precipitation of Thorium as Phosphate.*—2, 5, and 50 mgrms. Th as nitrate were treated by *P. 65*b*; white gelatinous precipitates separated on the addition of Na_2HPO_4, the precipitate being small and forming slowly in the experiment with 2 mgrms. Th. In the experiment with 5 mgrms. a 10 cc. portion of HF (1 volume 45 per cent HF to 2 volumes water) was poured several times through the filter; the solution was evaporated with H_2SO_4 to fuming, cooled, diluted, and excess NH_4OH added; no precipitate separated at once, and only a very small one on standing an hour.

*P. 65*b*, N. 4: *Behaviour of Manganese, Cobalt, Uranyl, and Vanadyl Salts in the Tests for Titanium and Zirconium.*—100 mgrms. Mn as $MnCl_2$ were treated by *P. 65*b*—*c*; with H_2O_2 no colour resulted, and no precipitate formed when Na_2HPO_4 was added, nor on standing one hour. After the addition of just sufficient powdered Na_2SO_3 to reduce the H_2O_2 (determined by testing portions of the solution with Ti solution), no precipitate formed, but when about 1 grm. more Na_2SO_3 was added a large precipitate separated. The experiment was repeated separately with 100 mgrms. Co as $CoCl_2$ and with 5 mgrms. U as $UO_2(NO_3)_2$; there was no change of colour with H_2O_2 and no precipitate with Na_2HPO_4 on adding enough Na_2SO_3 to destroy the H_2O_2. The experiment was repeated with 100 mgrms. U as $UO_2(NO_3)_2$; the solution was distinctly yellow before the H_2O_2 was added, and a large white precipitate formed on adding just sufficient Na_2SO_3 to decompose the H_2O_2.

10 and 100 mgrms. V as Na_3VO_4 were treated in separate experiments by *P. 65*b*—*c*; with the 10 mgrms. the colour obtained on adding H_2O_2 was similar to that obtained with 3 to 5 mgrms. Ti; with the 100 mgrms., however, the colour was of a much redder shade than that with titanium; on the addition of an excess of Na_2SO_3 the colour changed at once to blue, showing the presence of a vanadyl salt, and no precipitate had separated in either case after several hours in the cold or on boiling.

*P. 65*c*, N. 1: *Behaviour of Titanium in Acid Solutions towards Sodium Phosphate.*—To a solution containing 0·5 mgrm. Ti as chloride and 2 cc. H_2SO_4 (1·20) in 10 cc. were added 10 cc. 7 per cent $Na_2HPO_4 \cdot 12H_2O$ solution; the solution remained clear for ten minutes, but had become distinctly turbid in half an hour. Several solutions, containing 10 mgrms. Ti as chloride and varying amounts of H_2SO_4 (1·20) were treated in the same way; the flocculent precipitates were filtered off after ten minutes, and the filtrates were made alkaline with NH_4OH; in the experiment with 2 cc. acid the filtrate contained 0·5 to 0·1 mgrm. Ti; in that with 3 cc. acid, 1 to 3 mgrms. Ti; in the experiment with 5 cc. acid 3 to 4 mgrms. Ti.

10 mgrms. Ti as chloride were treated by *P. 65*b*—*c*, and after half an hour the mixture was filtered, and the filtrate made alkaline with NH_4OH; a very small precipitate was obtained containing about 0·5 mgrm. Ti. The experiment was repeated with 0·5 mgrm. Ti; the solution became distinctly turbid on decolorising with H_2SO_3. Half of the turbid solution was heated to boiling; a distinct, flocculent precipitate was obtained. The other half was allowed to stand in the cold for half an hour; the precipitate in this case also became somewhat flocculent.

*P. 65*d*, N.: *Test for Thallium with* KI.—0·5, 0·2, and 0·1 mgrm. Tl as Tl_2SO_4 were treated by *P. 65*d*, the total volume being about 10 cc.; a distinct yellow finely divided precipitate of TlI was obtained in each experiment, even in that with 0·1 mgrm. Tl. For proof that 500 mgrms. ferric iron does not interfere with the test, see T. A., No. 174—6.

*P. 65*d*, N. 3: *Flame Test for Thallium.*—0·5, 0·2, and 0·1 mgrm. Tl were precipitated by *P. 65*d* as TlI. The precipitates were collected on small filters and washed twice with a very little water. The moist filter was removed from the funnel, a looped platinum wire was drawn across its surface to collect a little of the precipitate, and introduced into a colourless gas flame; with 0·5 mgrm. Tl the momentary green colour was generally seen, but it was sometimes obscured by the yellow flame, due to sodium and to small fibres of paper; with 0·2 and 0·1 mgrm. Tl the green colour could not be detected with certainty. The experiments were repeated, except that the TlI precipitates were collected on hardened filters; the green flame was much more brilliant with 0·5 and 0·2 mgrm. Tl than in the corresponding experiments with ordinary filter-paper; it could, however, scarcely be seen with 0·1 mgrm. Tl.

P. 69, N. 1: *Potassium Cobaltic Nitrite.*—See Fisher (*Pogg. Ann.*, 1848, lxxiv., 115; Sadtler (*Am. Journ. Sci.*, 1870, [2], xlix., 196); Rosenheim and Koppel (*Zeit. Anorg. Chem.*, 1898, xvii., 35).

P. 69, N. 2: *Precipitation of Cobalt with Potassium Nitrite.*—0·1 and 0·3 mgrm. Co as $CoCl_2$ were treated by P. 69; in each experiment the solution became distinctly turbid within five minutes. The experiment was repeated in the absence of cobalt; the solution remained perfectly clear. For the detection of 0·5 mgrm. Co in the presence of 250 mgrms. Ni in P. 69, see T. A., 127.

500 mgrms. Co as nitrate were treated by P. 69; the mixture was shaken well, allowed to stand, and filtered after about half an hour, and again allowed to stand; a considerable precipitate again separated. The experiment was repeated except that the mixture was heated on a water-bath to 50 or 60° with frequent shaking for half an hour; it was allowed to cool, and filtered; no precipitate separated in the filtrate even on standing over night.

Separation of Nickel from Cobalt with Potassium Nitrite.—500 mgrms. Co and about 5 mgrms. Ni as nitrates were treated by P. 69; after standing twenty hours the mixture was filtered; the filtrate was evaporated almost to dryness with HCl, and made alkaline with NaOH; only a very small green precipitate separated corresponding to not more than 2 mgrms. Ni. Half of the cobalt precipitate obtained was treated by P. 70; a very good test for nickel was obtained. The experiment was repeated except that the mixture was heated on a water-bath for half an hour; less nickel was found in the filtrate than in the preceding experiment.

Precipitation of Potassium Nickelous Nitrite. — 250 mgrms. Ni (previously freed from cobalt by a KNO_2 treatment) were treated by P. 69, except that the volume was cut down to 50 cc. without, however, altering the total amount of reagents used; a distinct reddish coloured precipitate separated within twenty minutes which was proved to contain nickel but no cobalt by the borax bead test. For the action of HNO_2 on nickel salts and the formation of $K_4Ni(NO_2)_6$, see Lang (*Journ. Prakt. Chem.*, 1862, lxxxvi., 299); Hampe (*Lieb. Ann.*, 1863, cxxv., 346); and Reichard (*Chem. Zeitung.*, 1904, xxviii., 479, 885, 912).

P. 70, N. 2: *Delicacy of Hypobromite Test for Nickel.* —150 mgrms. Co free from nickel were treated by P. 70; no precipitate was observed, not even on filtering. The experiment was repeated except that 0·15 mgrm. Ni as $NiCl_2$ was added; the solution became dark coloured on adding excess NaBrO, but no precipitate collected; it was easily seen, however, on the filter. 0·2 mgrm. Ni as $NiCl_2$ was treated by P. 70; a distinct precipitate was obtained on filtering.

Separation of Nickel from Cobalt by Hypobromite.— 200 mgrms. Co and 0·5 mgrm. Ni as chlorides were treated by P. 70; the precipitate was tested for cobalt in the borax bead; no blue colour was obtained.

A large number of experiments were performed to determine the proper conditions for making this separation. In working with cobalt free from nickel, it was found that a precipitate of $Co(OH)_3$ always formed when the NaBrO reagent (or bromine water and NaOH) was added very soon after the addition of KCN, and that the necessary interval of time was greatly shortened by increasing the excess of KCN added.

The following experiments show that excess of NaBrO is essential to the precipitation of nickel. 1 mgrm. Ni as $Ni(NO_3)_2$ was treated by P. 69; the NaBrO solution was added in small portions, and after the addition of each portion the solution was tested with the starch KI paper; as long as this paper remained colourless no precipitate of $Ni(OH)_3$ separated, but after the precipitate formed the paper became blue when dipped into the mixture. The experiment was repeated with 10 mgrms. Ni. As long as the precipitation was incomplete the paper remained colourless, or only a small brown ring was formed on the paper, but after complete precipitation all of the paper immersed in the solution became brown or blue.

P. 70, N. 4: *Action of H_2S on Alkaline Tartrate Solutions containing Nikel or Cobalt.* — Villiers (*Comptes Rendus*, 1894, cxix., 1263; 1895, cxx., 46) found that when H_2S was passed into a NaOH containing freshly precipitated $Ni(OH)_2$ (but no tartrate) the hydroxide was quickly converted into black nickel sulphide, but that a portion of the nickel passed into solution giving a deep brown colour, proving that the presence of tartrate is not essential for the formation of the brown solution. 0·5 mgrm. Ni as nitrate was treated by both parts of P. 70; a deep brown solution was obtained on saturating the alkaline tartrate solution in a test-tube with H_2S. 0·1 and 0·2 mgrm. Ni as nitrate were dissolved in a little HNO_3 and treated by the second paragraph of P. 70, about 5 cc. of 10 per cent tartaric acid and 5 cc. excess of NaOH being added; clear dark yellow solutions were obtained on saturating with H_2S. The experiment was repeated with 1 mgrm. Ni; on passing in H_2S the solution remained nearly colourless for about one

minute, but finally a clear dark brown solution resulted. The experiment was repeated with 20 mgrms. Ni; on saturating with H_2S the liquid in the test-tube was opaque and almost black in colour. The liquid was filtered; very little precipitate remained on the filter. It was allowed to stand several hours; a black precipitate separated but the filtrate was still black and opaque.

20 mgrms. Co as $CoCl_2$ in a little dilute HNO_3 were treated by the second paragraph of P. 70; a black precipitate separated as soon as the H_2S was led in and the cobalt was completely precipitated within one minute. The mixture was filtered, the filtrate was saturated with H_2S, and the test-tube corked and set aside; the solution remained colourless for several hours. A similar solution was exposed to the action of the air in an open flask; it became dark yellow in about one hour owing to oxidation of the sulphide and consequent formation of polysulphide. The experiment was repeated except that 1 mgrm. Ni as $NiNO_2$ was also present; the excess of 10 per cent NaOH was 4 or 5 cc.; H_2S was led into the solution for about one minute and the CoS filtered off; the filtrate was nearly colourless. This was saturated with H_2S; it became brown, the colour being such as to indicate that very little of the nickel had been carried down with the cobalt. This experiment was repeated except that the H_2S was led through the solution for five minutes before the CoS was filtered off; the filtrate was of a lighter brown than before, indicating that over half the nickel had been carried down with the cobalt. The experiment was repeated except that the CoS was not filtered off till after ten minutes; the filtrate was almost colourless, and remained so on saturating again with H_2S. The experiment was repeated, the CoS being filtered off after half an hour; the filtrate was colourless and contained no nickel. In a similar series of experiments in which a smaller excess of NaOH than 4 to 5 cc. was added, the filtrate was light brown after one minute, and nearly colourless after five minutes, thus showing that there is more danger of losing nickel when the excess of alkali is small, in which case the brown solution is formed more quickly. This result that NiS is deposited on CoS after the separation of the latter was confirmed by several experiments.

Similar experiments were made with a mixture of 20 mgrms. Fe as $FeCl_3$ and 1 mgrm. Ni, and with one of 20 mgrms. Mn and 1 mgrm. Ni; good tests for nickel were obtained in both cases.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 3rd, 1908.

Sir WILLIAM RAMSAY, K.C.B., F.R.S., President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. Herbert Edwin Cocksedge, B.A., 5, Romola Road, Herne Hill, S.E.; Frederick Percy Dunn, B.Sc., Haresfield, Chorley Wood; Frederic Ferraboschi, B.A., Clare College, Cambridge; Walter Norman Haworth, M.Sc., Dacca House, Chorley; Eric Hayward, care of W. R. Criper, Esq., Konnager House, Konnager, near Calcutta; Robert Ernest Jackson, York Villa, Shepherd's Lane, Dartford; John Hugh Jeffery, 10, Daysbrook Road, Streatham Hill, S.W.; Joseph Allen Pickard, B.Sc., 3, West Kensington Terrace, W.; David Segaller, 69, Dover Road, Wanstead Park, E.; Reginald Wells Varley, 12, Mill Hill Road, Derby.

A ballot for the election of Fellows was held, and the following were declared duly elected:—Noel Charles Akers; Thomas Boles Allen, M.A.; Thomas Aubertin, B.A.; Maung Ba, B.A.; Thomas James Baker, D.Sc.; Maurice Barrett; Prince Mom Chow Bhunsri; Robert Bickerstaffe; William Colet Birch, B.A.; William Sudderick Brakes; Leonard Clement, B.A.; Robert Charles Cowley; Hugh Cecil Duckworth; Horace Barratt Dunnicliff, B.A., B.Sc.; Reginald Freeman Easton; Charles Everitt, M.A.; Henry Fairburn; Arthur Frank Girvan, B.Sc.; Henry Hamilton Green; John Griffin, B.A.; John Henry Albert Hebron, B.Sc.; Edward Hinks, B.Sc.; Joseph Albert Hughes; Thomas Ernest Hull; James Colquhoun Irvine, D.Sc., Ph.D; Grove Johnson; Patrick Anderson Keiller, B.Sc.; Joseph Kenyon, B.Sc.; H. V. Krishnayya, B.A.; Frank William Linch; Arthur Stanley Lindley; Gregor MacGregor, M.A., B.Sc.; Frederick Martin Kay Menzies, M.D., F.R.C.P.; William Herbert Miles; Charles James Millar; Edward Pigott Minett, M.D., M.R.C.S., L.R.C.P.; Howard Houlston Morgan, B.Sc.; Cyril Lawrence Norman; Alexander Robert Normand, M.A., B.Sc.; George Edward Pearson; John William Pearson, B.A.; Frank Perry; George O'Brien Power; Alexander Rennie; Ezra Lobb Rhead; William Frothingham Roach, M.D.; Maurice Salomon Salomon; Maung U. San; William Saunderson, B.Sc.; Herbert M. Shilstone, B.Sc.; Ernst Sommerfeldt, Ph.D.; Francis George Henry Tate; Frank Moreton Thomas; Sidney Gilbert Walton; John Watt, M.A.; Robert Dickie Watt, M.A., B.Sc.; Arthur Weighell; Frederick Williams, B.A.; William George Winterson, B.Sc.; William Ord Wootton, B.Sc.; Frederick Palliser Worley, M.A., M.Sc.; Allister McLean Wright.

Of the following papers, those marked * were read:—

*251. *Double Salts of Potassium Iodide with Mercuric Iodide and Dimercuriodocamphor in Organic Solvents.*" By JAMES ERNEST MARSH and ROBERT DE JERSEY FLEMING STRUTHERS.

When an aqueous solution of compound K_2HgI_4 is shaken with ether, ethyl formate, or some other organic solvents, the salt, $KHgI_3$, is extracted; it separates from its solution in ethyl formate as a heavy oil. In no case was the original salt, K_2HgI_4, dissolved. With camphor, the *double* salt, $KHgI_3,4C_{10}H_{16}O$, was obtained from dilute alcohol in light yellow gritty crystals melting at 65°.

Potassium iodide and dimercuriodocamphor, $C_{10}H_{14}OHg_2I_2$, also form a *double* salt readily soluble in acetone or alcohol, although the two constituents are separately very sparingly soluble.

*252. "*The Action of Mercuric Iodide on Ketones in Alkaline Solution.*" By JAMES ERNEST MARSH and ROBERT DE JERSEY FLEMING STRUTHERS.

Acetone reacts with mercuric iodide in cold aqueous potassium hydroxide according to the equation:—

$$C_3H_6O + 8HgI_2 + 6KHO = C_3OHg_5I_4 + 3K_2HgI_4 + 6H_2O.$$

Diethyl ketone reacts mainly according to the following equation:—

$$(C_2H_5)_2CO + 6HgI_2 + 4KHO = (CH_3{\cdot}CHg_2I_2)_2CO + 2K_2HgI_4 + 4H_2O.$$

Acetophenone reacts as follows:—

$$C_6H_5{\cdot}CO{\cdot}CH_3 + 6HgI_2 + 4KHO = HgI,C_6H_5{\cdot}CO{\cdot}CHg_3I_3 + 2K_2HgI_4 + 4H_2O.$$

The ketone mercury compounds form yellow precipitates. Both the yield and the composition of the precipitates agree with the equations given. In the case of acetone and diethyl ketone, standard alkali was used, and the amount converted into potassium iodide was determined. This amount also agrees with the above equations. The reaction thus affords a quantitative method for estimating the amount of ketones, either by weighing the precipitate formed, or by determining volumetrically the amount of alkali neutralised in the reaction.

*253. "*The Condensation of Camphor with Mercuric Iodide.*" By JAMES ERNEST MARSH and ROBERT DE JERSEY FLEMING STRUTHERS.

The following series of mercury camphor derivatives has been obtained:—

(A) $(C_{10}H_{15}OHg)_2O$, and its salts; (B) $C_{10}H_{14}OHg_2I_2$; (C) $(C_{10}H_{14}O)_2Hg_3O$; (D) $(C_{10}H_{14}O)_3Hg_4I_2$; (E) $(C_{10}H_{14}O)_4Hg_5I_2$; (F) $(C_{10}H_{14}O)_5Hg_6I_2$.

Of these compounds, B, D, E, and F are formed directly by the action of mercuric iodide on camphor in the presence of alkali. B is formed by the action of potassium ethoxide in alcoholic solution on camphor and mercuric iodide in the cold.

D is formed in a hot aqueous solution of potassium hydroxide containing excess of potassium iodide.

E is formed in a similar way with just sufficient potassium iodide to retain the mercuric iodide in solution.

F is formed when there is a defect of potassium iodide.

B is formed from D or E by the prolonged action of hot potassium hydroxide until all the iodine is removed.

Glacial acetic acid converts D into the *compound*, $C_{10}H_{14}OHg_2I_2$, which is insoluble, and the acetate of mercury camphor, which dissolves. From the latter, the base A is separated by the action of sodium hydroxide. The base forms salts, of which the *benzoate*, *sulphate*, *chloride*, *bromide*, and *iodide* have been prepared.

DISCUSSION.

Dr. M. O. FORSTER inquired whether definite products had been obtained by the dry distillation of these derivatives of camphor, some of which might be expected to yield dehydrocamphor; also whether any therapeutic value had been observed in connection with them.

Mr. MARSH replied that the compound $C_{10}H_{14}OHg_2I_2$ could be sublimed without decomposition, and that the other compounds decomposed, but the products formed had not been examined.

The material used in the experiments was the ordinary dextrorotatary camphor; the therapeutic and physiological action of the compounds had not yet been investigated.

*254. "*The Relation between Absorption Spectra and Chemical Constitution.* Part XIII. *Some Pyrones and Allied Compounds.*" By EDWARD CHARLES CYRIL BALY, JOHN NORMAN COLLIE, and HERBERT EDMESTON WATSON.

The absorption spectra of dimethylpyrone, chelidonic acid, xanthochelidonate, ethyl diacetylacetone, and several other substances have been examined in considerable detail. In many cases the spectra vary with the solvent used, and consequently it seems probable that these compounds can exist in more than one form. Diacetylacetone in particular may be either an open-chain compound or one with a ring formation.

The pyrones and their simpler derivatives appear to contain an oxygen bridge, the atom of oxygen in the ring being quadrivalent. This bridge is broken by the addition of a sufficient quantity of alkali, intermediate compounds being sometimes formed.

*255. "*Organic Derivatives of Arsenic.* Part I. *Dicamphorylarsinic Acid.*" By GILBERT T. MORGAN and FRANCES M. G. MICKLETHWAIT.

Dicamphorylarsinic acid, $HO{\cdot}OAs(C_{10}H_{15}O)_2$, colourless prismatic crystals, m. p. 266°, $[\alpha]_D$ 186·6°, is precipitated by acids from an alkaline extract of the product of the interaction of arsenious chloride and sodium camphor in toluene. Its *silver* salt, $AgO{\cdot}OAs(C_{10}H_{15}O)_2$, is a white, sparingly soluble substance, whereas its alkali salts dissolve readily either in water or alcohol. The acid is hydrolysed into camphor (2 mols.) and alkali arsenate (1 mol.), but only when its solution in aqueous caustic alkalis is evaporated almost to dryness.

When treated with phosphorus pentachloride, the acid gives rise to a *chloride*, from which the *anilide* may be obtained.

The authors are engaged in a study of the reactions occurring between the chlorides of the non-metals and organic compounds containing the characteristic group $CH_2{\cdot}CO$.

DISCUSSION.

Dr. LOWRY said he had gathered from the title that the compounds to be described were derivatives of camphoric acid. The use of the term "camphoryl" in this sense had been rendered inevitable by the analogy of "acetyl," "benzoyl," "carbonyl," &c., and had been so generally recognised that no alternative name had hitherto been suggested. The use of the same term for the camphor radicle $C_{10}H_{15}O$ was bound to lead to confusion, and it was very desirable that some distinguishing name should be adopted for this group. Camphor derivatives had usually been named by means of prefixes, but the case of "camphocarboxylic acid" suggested an alternative usage: could not the arsenic compound be described by an analogous name as "dicamphoarsenic acid"?

Dr. M. O. FORSTER defended the use of the expression "camphoryl" for the complex $C_{10}H_{15}O$, and invited Dr. Lowry to formulate "dicamphorylarsinic acid" on his assumption that camphoryl should stand for the radicle of camphoric acid.

In reply to Dr. Pyman, Dr. MORGAN said that they had not hitherto obtained the corresponding arsonic acid from the products of this condensation. With reference to the question of nomenclature raised by Dr. Lowry, it seemed to the authors that the prefix "camphoryl" was a suitable one to use for this acid, in which opinion they had the support of other investigators of camphor derivatives.

*256. "*Tellurium Dicyanide.*" By HERBERT EDWIN COCKSEDGE.

When tellurium tetrabromide and silver cyanide are heated together for some days in boiling benzene, tellurium dicyanide is formed.

On extracting the residual silver salts with ether, and evaporating the ether at the ordinary temperature, white crystals of the composition $2Te(CN)_2,(C_2H_5)_2O$ are obtained. By heating these and subliming the product in a vacuum, the pure *dicyanide*, $Te(CN)_2$, results in the form of white crystals.

The compound is partly decomposed by heat, giving cyanogen and tellurium. It is at once hydrolysed by water, with precipitation of black flocks of tellurium.

*257. "*Boron Thiocyanate.*" By HERBERT EDWIN COCKSEDGE.

Boron thiocyanate, $B(SCN)_3$, is prepared by the action of boron bromide on silver thiocyanate in cold benzene solution. On evaporation of the solvent at the ordinary temperature, colourless crystals remain.

The compound resembles the corresponding thiocyanates of phosphorus and silicon in general reactions, but decomposes readily on heating. The action of aniline on the compound in benzene solution points to the thiocyanate constitution.

*258. "*The Viscosity of Fuming Sulphuric Acid.*" By ALBERT ERNEST DUNSTAN and ROBERT WILLIAM WILSON.

The authors have investigated the viscosity-concentration curve afforded by various mixtures of sulphuric acid and sulphur trioxide. They find a maximum point at the concentration corresponding with H_2SO_4,SO_3.

DISCUSSION.

Dr. SENTER said that the maxima in the curves for binary mixtures obtained by plotting viscosity against composition were usually somewhat difficult to interpret. Both sulphuric acid and water appeared to exist as very complex molecules in the pure state, and it was probable that both of them were in a simpler molecular condition when mixed. He wished to ask Mr. Dunstan how, in his opinion, the viscosity curve would be affected by a progressive simplification of the molecules of the components on dilution. If it was assumed that, owing to the increased surface of the particles, the effect of this simplification was to increase the viscosity, the maximum in certain binary mixtures of associated liquids (for example, alcohol and water) could be accounted for without assuming that the components enter into chemical combination. On the other hand, independent evidence pointed to the existence of chemical compounds in mixtures of sulphuric acid and water, and the latter system was therefore very complicated.

Mr. Dunstan, in reply to the President, agreed as to the advisability of determining the molecular surface energy of the mixture, the composition of which approximated to H_2SO_4,SO_3; in answer to Dr. Senter, he said that there were two explanations to the maximum points in viscosity-concentration curves; Jones and Veazey (*Am. Chem. Journ.*, 1907, xxxvii., 405) have asserted that such maxima are due to dissociation; that is, by the proportionally larger frictional surfaces of smaller molecular masses, but he himself contended that in the first place the maxima correspond with aggregates of simpler molecular composition, that such aggregates have actually been isolated, and that heat effects and many other physical properties attain their maxima at the same points. The argument as to larger frictional surfaces only held good if it were definitely known that the aggregates are regular in shape and smooth in surface.

Moreover, since associated substances were proportionately more viscous than non-associated compounds, he considered that the action of the sulphuric acid on the water (and *vice versâ*) was (1) simplification of the homogeneous aggregates, and then (2) association of these products.

259. "*The Decomposition of Carbon Dioxide by the Silent Electric Discharge.*" By Alfred Holt, jun.

Carbon dioxide, which had been carefully dried by phosphoric oxide, was found to be decomposed to an amount which increased as the pressure of the gas diminished. About 48 per cent decomposition was obtained under 30 mm. pressure, and about 3 per cent decomposition under 700 mm. pressure.

The amount of decomposition was found to vary with the degree of dryness of the gas, the intensity of the discharge, and the form of ozoniser employed, but in all cases diminished pressure gave greater decomposition.

Moist carbon dioxide was found to decompose to a larger extent as the pressure of the gas increased, ranging from 3 per cent decomposition under 50 mm. pressure to about 14 per cent under 700 mm. pressure. The weaker the discharge the less gas was decomposed at all pressures.

Comparison of these results with those obtained when moist and dry carbon dioxide is decomposed by ultra-violet light, or induction sparks, leads to the conclusion that the silent discharge at low pressures acts chemically, mainly like ultra-violet light, whilst as the pressure of the gas increases, it behaves more and more as though it consisted of actual sparks.

260. "*Note on Oxalyl Chloride.*" By Humphrey Owen Jones and Hubert Sanderson Tasker.

The authors' investigations on the properties and reactions of oxalyl chloride had been in progress some months when a paper on the same subject was published by Staudinger (*Ber.*, 1908, xli., 3558). It was found, in agreement with Staudinger, that the yield of oxalyl chloride obtained by Fauconnier's method, namely, by the action of phosphorus pentachloride on ethyl oxalate, is very poor, but the yield can be enormously increased by carrying out the reaction at 160° under pressure.

It was found impossible to free the oxalyl chloride entirely from phosphorus compounds by distillation, as the process is rendered more difficult by the presence of *as*-dichloroethane among the products. The amount of phosphorus trichloride formed is small, and the chief product is phosphoryl chloride; ethyl chloro-oxalate and ethyl trichloroacetate are formed in large quantities.

The major portion of the oxalyl chloride distils at about 70°, and then contains about 3 per cent of phosphoryl chloride. Oxalyl chloride reacts vigorously with water, and yields some oxalic acid, together with carbon monoxide and carbon dioxide, whereas Staudinger states that oxalic acid is only obtained when the vapours react.

Oxalyl chloride reacts vigorously with primary and secondary amines to produce substituted oxamides, whereas with tertiary amines it forms additive products. The *additive* compound with pyridine, $C_2O_2Cl_2,2C_5H_5N$, is a white solid, which reacts with water to give pyridine hydrochloride with carbon monoxide and dioxide, and no oxalic acid; it reacts with primary or secondary bases to give pyridine hydrochloride and substituted oxanilides. This is a better method of preparing these substances than by using the chloride direct. The compounds formed with tertiary amines resemble closely the corresponding compounds obtained from carbonyl chloride.

The authors are at present engaged in examining the reactions of oxalyl chloride on a number of substances, chiefly derivatives of organic metallic compounds.

261. "*The Densities of Krypton and Xenon.*" By Richard B. Moore.

The author has prepared pure xenon and krypton from the residues from 120 tons of liquid air. The density of xenon was found to be 65·35, and that of krypton 41·506, the atomic weights being, on the assumption that the gases are monatomic, 83·012 and 130·70 respectively (O = 16).

262. "*The Action of Reducing Agents on Tannic and Gallic Acids.*" By Walter Myers Gardner and Herbert Henry Hodgson.

The object of the work was to devise a rapid and accurate method for the estimation of tannic and gallic acids, and it was thought that a quantitative yield of benzoic acid might be obtained on reduction. Previous work on the action of reducing agents on these acids had been published by Böttinger (*Annalen*, 1890, cclviii., 252) and Guignet (*Comptes Rendus*, 1891, cxiii., 200). The former obtained no reduction products, but using different conditions Guignet found that both tannic and gallic acids were converted into benzoic acids.

The authors have repeated Guignet's work, but obtained a very poor yield of benzoic acid. In the further work, the following were tried as reducing agents:—Zinc, sodium, magnesium, aluminium, sodium hyposulphite, and hydriodic acid; experiments being made in most cases under acid, alkaline, and neutral conditions. The general results were that tannic and gallic acids are little affected by neutral or acid reducing agents, but that they react readily in alkaline solution, In no case, however, was a quantitative yield of benzoic acid obtained, and this was shown to be due to the slow destruction of benzoic acid by alkaline reducing agents.

The original object of the investigation was therefore not attained, but several interesting observations were made.

263. "*The Action of Iodine on Phenols and a Modified Process for the Estimation of Tannic Acid.*" By Walter Myers Gardner and Herbert Henry Hodgson.

In the preceding paper, the authors have shown that tannic acid in alkaline solution rapidly reacts with reducing agents, and, based on this observation, a quick and accurate method for the estimation of tannic acid has been developed. The process is carried out as follows:—To an aqueous solution of tannic acid, standard iodine solution is added in excess. Aqueous sodium hydroxide is then added drop by drop until the colour due to the iodine disappears, after which concentrated hydrochloric acid is added in sufficient excess to precipitate the unabsorbed iodine, the amount of which is estimated by standard sodium thiosulphate. Two estimations are necessary in the case of commercial tannins. Firstly, that of the total iodine absorbed, and secondly, that of the iodine absorbed after removing the tannic acid from the solution by precipitation with gelatin; the difference giving the iodine absorbed by the tannic acid. Previous work by Messenger and Nortmann (*Ber.*, 1890, xxiii.; 2753), F. Musset (Chemical News, 1885, li., 42), and A. Moullade (*Journ. Pharm. Chim.*, 1905, xxii., 153) on somewhat similar lines has been repeated, and it has been further shown that the process is available for the estimation of many phenols; for example, phenol, catechol, quinol, salicylic acid, pyrogallic acid, and gallic acid, in addition to tannic acid. With these substances, the amount of iodine absorbed is in direct ratio to the number of hydroxyl groups present, one

hydroxyl group requiring one molecule of iodine; but with other phenols the reaction, although quantitative, is empirical. The presence of other groups, such as NO_2, NH_2, OMe, as well as the position of the hydroxyl groups in the benzene nucleus, was found to have a determining influence on the course of the reaction. Following Wijs's investigations on the action of Hübl's solution, the authors think that the active substance is the hypoiodate formed, but they are further investigating this assumption.

264. "*Ester Catalysis and a Modification of the Theory of Acids.*" By Edward Fitzgerald and Arthur Lapworth.

The previous observations on esterification and ester hydrolysis in acetone already referred to (*Proc.*, 1908, xxiv., 152) have been confirmed and extended.

If it be assumed that hydrogen ions are responsible for acid catalysis in esterification and ester hydrolysis, the authors find it possible to explain the facts on one proposition only, namely, that the addition of water to a solution of an acid in acetone or alcohol greatly diminishes the number of hydrogen ions, the alternative theories—that the intermediate products are shared between the alcohol and water, or the view applied by Arrhenius to the hydrolysis of salts of weak bases—having proved inadequate.

It would then appear that the proportion of hydrogen ions in an aqueous solution of hydrogen chloride must be extremely small, and this makes it seem probable that they are not altogether indispensable to a satisfactory theory of acids.

The hydrated hydrogen ion or hydroxonium ion, H_2O, often assumed to be the conducting positive ion in aqueous solution of acids, is, in this conception, inert as a catalyst, so that these suggestions are not only at variance with the Arrhenius-Ostwald view of connection between conductivity and reactivity, but are essentially opposed to theories of hydrion catalysis in which the hydroxonium ion is regarded as catalytically active (compare Goldschmidt and Udby, *Zeit. Physik. Chem.*, 1907, lx., 731).

In most physical measurements the hydrogen ions proper may be neglected, and the above theory virtually takes the form that the anticatalytic effect of water on catalysis by acids is due to the fact that water in the same sense as ammonia is a more powerful base than the solvent, and, usually, the substance catalysed, and diminishes the available free acid by converting it into hydroxonium salt.

265. "*An Examination of the Conception of Hydrogen Ions in Catalysis, Salt Formation, and Electrolytic Conduction.*" By Arthur Lapworth.

Small quantities of water depress the catalytic effect of acids on the bromination of acetone and on the hydrazobenzene conversion in alcohol and other solvents in just the same way as in the case of esterification (Goldschmidt) and the decomposition of ethyl diazoacetate (Bredig). Water, to the extent of 0·5 per cent in acetone itself, depresses the velocity with which it absorbs bromine under the influence of acids to considerably less than one-half of its original value.

Proceeding from the most generally accepted view that free hydrogen ions are responsible for catalysis by acids, a strict mathematical expression applying to the state of an acid in a medium is easily developed. By means of this it is not difficult to show that all the generalisation of Ostwald and Arrhenius applying to the variations of conductivity and catalytic activity with volume and the measured degree of dissociation will be as true when the medium consists of, or contains, a mixture of bases as when it is neutral; further, that all acids at infinite dilution in a given medium will exhibit the same equivalent catalytic activity. But this is mathematically true when the hydrogen ions in all cases are then made = 0.

In order to connect these conclusions with the basic properties of common oxygenated solvents, a simple indicator method has been devised. The phenomena observed with water on the indicator salt in alcohol, simulate closely the effects of water on acid catalysis. Using this process in several media, Mr. R. W. L. Clarke reports, as a first approximation to the real relative affinities of some weak bases; carbamide, 17; water, 1; ether, 0·12; ethyl alcohol, 0·06. The values are fairly concordant amongst themselves, and are of the expected order.

SOCIETY OF CHEMICAL INDUSTRY.

(London Section).

Ordinary Meeting, December 7th, 1908.

Dr. Lewkowitsch in the Chair.

"*Cellulose and Chemical Industry.*" By Messrs. Cross and Bevan.

This paper is a retrospect and a general forecast. Such a review is suggested by the synchronism of the date of the formation of the Society with the commencement of the wood-pulp (cellulose), and "artificial silk" or lustra-cellulose industries. In the generation which has elapsed, this latter industry has reached large dimensions; the current production is estimated at 5000 tons per annum. The authors examine the basis of competition of these artificial or "chemical" fibres, with silk one the one hand, and vegetable (fibrous) "mechanical" textiles on the other. With further refinements of the processes, and further constitutional modifications of the colloidal cellulose, which are already shown to be attainable, new avenues of expansion of the industry are assured. Problems of fundamental importance to the flax, jute, and wood-pulp industries are briefly discussed. Generally, there is considerable activity in the more special developments of the cellulose industries, and a very large number of definite problems are presented to the chemist for investigation and solution. Manufacturers cannot be expected to take long forecasts, or to devote means to research beyond what is necessary for attacking proximate problems connected with routine. Chemists must adapt themselves to the situation, and find their own ways and means for work in one of the most fruitful and attractive fields of investigation.

"*The Viscosity of Indiarubber and Indiarubber Solutions, with particular reference to its bearing on the Strength or 'Nerve' of Crude Rubber.*" By P. Schidrowitz and H. A. Goldsbrough.

The authors have studied the viscosity of different crude rubbers in benzene solutions, and have correlated the viscosity curves and values with the figures obtained by tension (breaking, strain, &c.) experiments. The viscosity curves show that, beyond a certain concentration (which the authors call the "critical-point"), the curve becomes a straight line, but that the straight lines so obtained from different rubbers are not directly comparable. It is necessary, therefore, for purpose of comparison, to calculate a value from two points by the ordinary equation $y = mx + c$. The results obtained appear to show a distinct connection between viscosity and "nerve," weak rubbers yielding a flat, strong rubbers a steep curve. Assuming that some of the peculiar physical attributes of rubber are due to de- or re-polymerisation, the viscosity method seems likely to afford a useful means of measuring the molecular condition, and in any case of distinguishing between that which the authors term "mechanical" strength and "physical or chemical" nerve.

"*The Structure of Crude Indiarubber.*" (Preliminary Note). By P. Schidrowitz, Ph.D.

The author states that his experiments appear to warrant the important conclusions that the globules are not destroyed by coagulation of the latex, and that they continue to exist in the dry rubber, and are not removed even by dissolving the latter in benzene. Microscopic examinations of benzene solutions by means of the dark ground illumination method discloses the presence of bodies which, in size and general appearance, are strikingly similar to the

original globules of the latex, and the same applies to the spherical bodies observed in dry rubber obtained by different methods, including the evaporation of solutions. The presence of the globules, the author points out, may explain some of the more important physical properties of crude and, perhaps, of vulcanised indiarubber.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlvii., No. 20, November 16, 1908.

Agricultural Use of Calcium Cyanamide.—A. Müntz and P. Nottin.—In experiments in which the quantities of cyanamide employed were ten to twenty times those which would be used in agricultural practice, the nitrifying oganisms appeared to be paralysed at first, and even a slight denitrification was observed. After a short time, however, the organisms became acclimatised, and nitrification proceeded normally. The decrease of activity is due to the cyanamide itself and not to the quicklime which it always contains. The results obtained in the nitrification experiments show that calcium cyanamide is one of the most active nitrogenous manures, being practically equivalent to ammonium sulphate. It can be applied either at the time of sowing or as a top dressing, but in the latter case it must be used only in rainy weather.

Volumetric Composition of Ammonia and Atomic Weight of Nitrogen.—Ph. A. Guye and A. Pintza.—The authors have determined the density of the gas N_2+3H_2, obtained from the decomposition of a given weight of NH_3, and have thence calculated the atomic weight of nitrogen. Taking O = 16 the atomic weight lies between 14·00 and 14·02, which confirms the value 14·01 adopted by the International Commission.

Fused Amosphous Alumina and the Production of Artificial Sapphires.—Louis Paris.—When oxides capable of giving a blue coloration are added to alumina fused before the blowpipe flame no coloration is produced. The oxides float on the surface of the alumina, without mixing with it or colouring it. If, however, the alumina used is not chemically pure but contains some hundredths of a foreign oxide, *e.g.*, lime, cobalt gives a blue coloration, and the optical properties of the alumina show that the latter is not crystalline but amorphous. The specimens of coloured alumina thus produced so closely resemble sapphires that even experts have been unable to distinguish them from the naturally occurring gems. With nickel yellow specimens have been obtained which rival the Ceylon sapphire.

Berichte der Deutschen Chemischen Gesellschaft. Vol. xli, No. 15, 1908.

Hydrofluorides of Weak Organic Bases.—R. F. Weinland and F. Reischle.—A saturated alcoholic solution of hydrofluoric acid reacts with acetanilide and *p*-ethoxyacetanilide, giving hydrofluorides of the composition Acetanilide + 1HF and *p*-Ethoxyacetanilide + 3HF + $2H_2O$. Similarly triphenylamine dissolves in saturated alcoholic strongly fuming hydrofluoric acid, and from the solution the monohydrofluoride slowly separates in the form of a pale green powder. It is not very stable, losing hydrofluoric acid in dry air. Diphenylamine gives di- and trihydrofluorides and aniline mono-, di, and tri-hydrofluorides. Azobenzene gives a compound which is fairly stable in a desiccator, and dimethylpyrone forms hydrofluorides, which are quite stable in dry air.

Molecular Condition of Fused Salts.—Richard Lorenz and F. Kaufler.—The similarity between the electrochemical behaviour of fused salts and solutions of salts suggests that they are in both cases in the same molecular condition, and the question arises how far fused salts conform to the laws of Arrhenius' theory for aqueous solutions. To decide this it is necessary to determine the molecular weights of the salts in the fused state. If it is true that the salt simply dissociates into its ions, the molecular weights observed must be lower than those which correspond to the chemical formulæ, as is the case with dilute aqueous solutions. If the molecular weight is greater complex ions must be formed and association must occur. The surface tension method was used to determine the molecular weights, and the results showed that salts in the fused state are complex compounds, the ions being complex ions, a result which seems to gain in probability when it is remembered that double salts such as those of lead chloride and alkali chloride, in which a complex anion of lead and chlorine is present, exist in a molten state.

Silicon Iodoform.—Otto Ruff.—Silicon iodoform may be obtained from silicon chloroform by converting the latter either into the nitrogen hydride SiNH by the action of liquid ammonia, or with aniline into the hydrotrianilide $SiH(NH.C_6H_5)_3$, and then treating these compounds with dry hydriodic acid in carbon disulphide or benzene solution. The second method gives the better yield, and is also more convenient. The nitrogen compound is similarly split up by hydrochloric and hydrobromic acids. Silicon iodoform is a colourless liquid which very readily forms silicon formic acid with water. It yields an oxyiodide with oxygen. It burns readily in air, and at a high temperature its vapour forms an explosive mixture with air.

Ferromagnetic Nitrogen Compounds of Manganese.—E. Wedekind and Th. Veit.—On heating manganese in a current of ammonia to the temperature of the oxyhydrogen blowpipe flame a relatively strongly magnetic preparation is obtained. The analysis of it gives results which correspond to the formula Mn_7N_2. It may be shown that whether this is actually a new compound or not the magnetisability of compounds of manganese and nitrogen increases as the amount of manganese increases. When powdered manganese is heated in dry hydrogen gas over a coal-gas blowpipe flame a molten mass is obtained which is feebly magnetic. The oxide obtained by heating manganese powder in a current of oxygen is not magnetic.

Spectrum of Neo-Erbium Oxide.—K. A. Hofmann and Günther Bugge.—The power of erbium oxide of forming a reflexion spectrum with sharp lines which are regularly grouped about definite maxima is not shared by the solid salts in general. The oxalate and the fluoride obtained by evaporation show bands similar to those of the dissolved nitrate or sulphate, although differences in intensity and position can be detected. With the dry sulphate lines appear as well as the bands, so that a transition from the band spectra of the salts to the line spectra of the oxide seems possible. Apparently the light-absorbing and emitting electrons of the erbium atom in the oxide perform far more regular oscillations than in compounds with acid residues or water molecules, and in the solid state the conditions of the freedom of the movement of the electrons are similar to those in dilute gases.

Constitution of Copper Acetylide.—Johannes Scheiber.—Copper acetylide exists in two forms, one anhydrous with the formula C_2Cu_2, and the other monohydrated $C_2Cu_2.H_2O$. The behaviour of the hydrated acetylide seems to harmonise best with the formula CH:C.Cu.Cu.OH, and Makowka's formula appears to be incorrect. Copper acetylide can be prepared in the pure state. The carbonaceous residue left when a preparation is dissolved in acids is not an impurity, but it is a humus-like substance the formation of which must be ascribed to secondary processes.

MEETINGS FOR THE WEEK.

TUESDAY, 29th. THURSDAY, 31st. SATURDAY, Jan. 2nd. } Royal Institution, 3. (Christmas Lectures, adapted to a Juvenile Auditory). "The Wheel of Life," by Prof. W. Stirling, M.D., &c.

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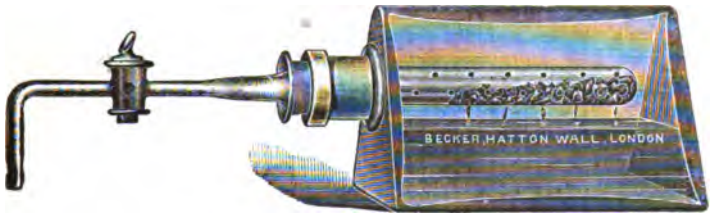

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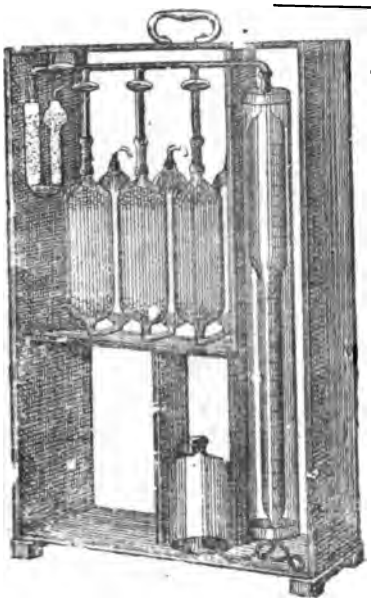

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" do. No. 4 " 0° C.
CARBOLIC ACID DISINFECTING POWDER.

CARBOLIC ACID GLYCERINE SOLUTIONS.
CRESYLIC ACID.
SULPHO-PHENIC ACID (Cryst.).
SULPHO-PHENATES & SULPHO-CRESYLATES OF SODA, POTASH, ZINC, IRON, and ALUMINA.

ACETONE.
WARDLE'S FERRICUM (Sewage Precipitant).
PICRIC ACID (Cryst. and Paste).
AURINE (Rosolic Acid), Cake and Solution.

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